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EVALUATION OF CORROSION INHIBITORS AS LUBRICITY IMPROVERS

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ABSTRACT (Continue on reverse if necessary and identify by block number) The technical effort described herein was directed at evaluating the corrosion inhibitors (CI) currently approved under the MIL-I-25017D qualified products list (QPL). The thrust of the effort focused on the following: (1) establishment of relative effective concentrations for approved CI, (2) generation of working curves to profile CI performance in jet fuels, (3) development of an approach for incorporating a lubricity requirement into MIL-I-25017, (4) refinement of the reverse phase high performance liquid chromatography (RPHPLC) method for determining CI content in jet fuels, (5) determination of applicability of the RPHPLC to WPL CI, and (5) generation of an RPHPLC spectral library of VPL CI in JP-4.			
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08 Psychology	08 Personnel selection, training and evaluation	14 Terrestrial magnetism	10 Acoustics
09 Psychology	09 Psychology (individual and group behavior)	15 Military Sciences	11 Crystallography
10 Psychology	10 Psychology	01 Antisubmarine warfare	12 Electricity and magnetism
11 Sociology	11 Sociology	02 Chemical, biological, and radiological warfare	13 Fluid mechanics
08 Biological and Medical Sciences	12 Escape, rescue and survival	03 Defense	14 Heat transfer
01 Biochemistry	13 Food	04 Antimissile defense	15 Hydraulics
02 Biengineering	14 Hygiene and sanitation	05 Intelligence	16 Information theory
03 Biology	15 Industrial (occupational) medicine	06 Logistics	17 Intercommunications
04 Biotics	16 Life support	07 Nuclear warfare	01 Missiles launching and ground support
05 Clinical medicine	17 Medical and hospital equipment and supplies	08 Operations, strategy, and tactics	02 Missile trajectories
06 Environmental biology	18 Environmental biology	09 Nuclear warfare	03 Missile warheads and fuzes
07 Food	19 Escape, rescue and survival	10 Operations, strategy, and tactics	04 Missiles
08 Hygiene and sanitation	20 Food	11 Air and space launched missiles	05 Telemetry
09 Industrial (occupational) medicine	21 Industrial (occupational) medicine	12 Navigation, Communications, Detection and Countermeasures	06 Nuclear warheads
10 Life support	22 Industrial (occupational) medicine	01 Acoustic detection	07 Operations, strategy, and tactics
11 Medical and hospital equipment and supplies	23 Industrial (occupational) medicine	02 Communications	08 Navigation and guidance
12 Microbiology	24 Industrial (occupational) medicine	03 Direction finding	09 Optical detection
13 Microbiology	25 Industrial (occupational) medicine	04 Electromagnetic and acoustic countermeasures	10 Radar detection
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01 Air conditioning, heating, lighting and ventilation	02 Civil engineering	02 Civil engineering	02 Spacecraft
02 Construction equipment, materials and supplies	03 Construction equipment, materials and supplies	03 Construction vehicles and ground support	04 Spacecraft support

FOREWORD

This report describes the technical effort conducted under Air Force Contract No. F33615-85-C-2508, entitled "Properties of Aircraft Fuels and Related Materials." All research conducted under this contract was administered under the direction of Mr. Steve Anderson, Project Engineer, Fuels Branch of the Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories (AFWAL) and Mr. Paul A. Warner, Program Manager, United Technologies Corporation, Pratt & Whitney (P&W).

The technical effort disclosed herein was performed during the period 16 February 1987 through 15 February 1988 under Task Order No. 13, entitled "Evaluation of Corrosion Inhibitors as Lubricity Improvers." Tedd B. Biddle was the P&W Task Order Manager.

The authors wish to thank Steve Anderson, the Air Force Project Engineer, for his assistance in providing technical direction and support in the many decisions required for the successful completion of this program; and Chuck Martel, AFWAL/POSF, for providing the historical background on corrosion inhibitors (CI), their use, and the rationale applied to the specification requirements directed at qualifying candidate CI.

The authors also wish to acknowledge and thank the following P&W personnel: Dick Meehan for overseeing the laboratory effort conducted under this task; Michael Polito for his diligence in the meticulous formulation of more than 576 fuel blends and well over 1,000 Ball-On-Cylinder Lubricity Evaluator (BOCLE) analyses; William Cellich for his assistance in the performance of the BOCLE tests and the plotting and careful interpretation of all polynomial curve fits; Donald Yost for his assistance in compiling and organizing test data; and Suzanne Guisinger whose computer programming skills permitted timely and efficient archiving and analysis of all test data.



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LIST OF ABBREVIATIONS

AFWAL	Air Force Wright Aeronautical Laboratories
BOCLE	Ball-On-Cylinder Lubricity Evaluator
CI	Corrosion Inhibitor
CRC	Coordinating Research Council
CT	Clay Treated
DLA	Dilinoleic Acid
g	Gram
g/m ³	Gram Per Cubic Meter
IEC	Ion Exchange Chromatography
IS	Ionization Suppression
k'	Capacity Factor
MAC	Maximum Allowable Concentration
MEC	Minimum Effective Concentration
mL	Milliliter
µL	Microliter
MM	Millimeter
MW	Molecular Weight
MLA	Monolinoleic Acid
nm	Nanometer
OC-ALC	Oklahoma City Air Logistics Command
ppm	Parts Per Million
P&W	Pratt & Whitney
QPL	Qualified Product List
R	Correlation Coefficient
REC	Relative Effective Concentration
RPM	Revolutions Per Minute
RPHPLC	Reverse Phase High Performance Liquid Chromatography
SE _n	Standard Error of Estimate
TLA	Trilinoleic Acid
UV	Ultraviolet
WSD	Wear Scar Diameter
WSI	Water Separometer Index
WSIM	Water Separation Index, Modified

SECTION I

INTRODUCTION

At present, the mechanisms associated with fuel lubricity are not well understood. However, it became apparent in the mid 1960s that corrosion inhibiting additives are responsible for imparting good lubricity characteristics to the fuel. The requirement for a corrosion inhibitor (CI) was rescinded at that time, resulting in numerous lubricity problems. The requirement for a CI was reinstated, and lubricity incidents were dramatically reduced. It is now generally accepted that the primary role of a CI is lubricity enhancement and not corrosion inhibition.

Fuel lubricity continues to receive considerable attention and concern in response to reports of lubricity related incidents. During the first eight months of 1986 alone, the U.S. Air Force experienced operational problems with 30 TF30 engine hydraulic fuel pumps in F-111 aircraft flying out of Cannon Air Force Base. The Oklahoma City Air Logistics Center (OC-ALC) investigated the incidents and determined that the problem was due to sensitivity of the pump to the lubricity of the fuel. The investigation also revealed that the same CI was used in each case. Addition of a different CI at the fuel terminal resulted in preventing further occurrences of excessive wear. No reports of pump failures have occurred since the change was made (Ref. 1).

Similar lubricity problems have been reported at other locations. In response to these incidents, the Air Force initiated a program to evaluate the CIs qualified under MIL-I-25017D for their effectiveness as lubricity enhancers in aviation turbine fuels. The intent of this effort was to modify the CI specification to include a requirement for lubricity. Currently, the use of fuel soluble CI is one solution to circumventing wear problems caused by fuels lacking natural lubricating agents.

1. BACKGROUND

An excellent historical background of the current requirement for the addition of CI in jet fuel was presented by Chuck Martel, et al in an Air Force Aero Propulsion Report published in July 1974 (Ref. 2). The technical report, entitled "Aircraft Turbine Engine Fuel Corrosion Inhibitors and Their Effects On Fuel Properties," outlines the initial specification requirements and the subsequent revisions that resulted in the required use of CI specifically for improving fuel lubricity.

The Air Force study reports that with the introduction of jet aircraft and kerosene type fuels in the mid 1940s, fuel contamination problems were experienced that were much more severe than previously experienced with aviation gasolines. The greater viscosity and density of jet fuels resulted in the entrainment of water and solid matter that often carried over into aircraft fuel systems. The addition of CI to jet fuels was begun in the early 1950s to combat excessive corrosion in ground fuel systems and subsequent carry over of corrosion products into the aircraft.

The first requirement for CI addition to JP-4 type fuels was by Amendment 1 to the MIL-F-5624B jet fuel specification in March of 1954. In October 1954, a specification for CI was issued as MIL-I-25017 and entitled "Inhibitor, Corrosion, for Aircraft Engine Fuels." Performance of a corrosion test was required by this specification to determine the effective level of CI to be added to a JP-4 fuel. The 20-hour test conducted at a bath temperature of 38°C (100°F) defined the minimum effective concentration required for each CI qualified to the specification.

The first QPL for CI was issued in September of 1955 as QPL-25017-1 and contained three approved inhibitors. The revisions to MIL-I-25017 that followed included Revision A in

September 1959 that classified the corrosion test designation to be ASTM Method D665, Procedure B. In May 1955, and again in December 1957, revisions were made to MIL-F-5624 (Revisions C and D) that dictated that a CI "shall be" added to JP-4 and JP-5 fuels. Revision E, however, was issued in March 1960 that changed this wording to "may be" added. Revision F of this specification followed in September 1962 and stated that a CI "shall be added to JP-4," but "shall not be added to JP-5 unless approval is obtained."

Despite the early success of CI to alleviate lubricity related problems, it was immediately apparent that CI were not without potential shortcomings.

In the late 1950s some CI caused severe fuel/water separation problems. In the presence of CI, fuel filter coalescer units failed to efficiently remove undissolved water from the fuel. The result was the removal of a number of CI from the QPL. A Water Separometer Index (WSI) limit was incorporated into the fuel specification to ensure acceptable fuel/water separation characteristics in the presence of additive.

In the early 1960s, jet fuel filtration problems were associated with the use of CI. The formation of a gelatinous material that rapidly plugged filters resulted from a chemical reaction involving undissolved water, metal (aluminum, steel, magnesium, or zinc) and CI. Consequently, the requirement for a CI to be added to JP-4 was deleted in November 1965. The revised specification stated that a CI shall not be added to grade JP-4 or JP-5 without prior approval from the end user (Refs 3 and 4).

The repercussions caused by the elimination of CI were immediate and readily apparent. A number of occurrences of fuel control malfunctions were reported. Ultimately these were traced back to the removal of the CI that was functioning as a lubricity agent in gas turbine fuels. Because of the severity of the problems, the Air Force issued an operational Technical Order in March 1966 to blend CI into all JP-4 fuel at the base level. Amendment 1 to MIL-T-5624G was issued in November 1966 reinstating the requirement for CI conforming to MIL-I-25017 to be blended into JP-4 by the supplier. The use of CI in JP-5, however, was excluded. The requirement for the addition of CI to JP-5 type fuel was not adopted until revision 'L' of MIL-T-5624 was issued in January 1983. Although not entirely resolved, fuel lubricity problems both domestically and abroad are currently controlled by the mandatory use of CI.

The current QPL contains 15 approved CI. Among these, DCI-4A, Nalco 5403 and ARCO IPC 4445 tend to dominate in Air Force usage. DCI-4A and Nalco 5403 are also used, as well as Unicor J, extensively by the U.S. Navy. Any of the 15 CI presently qualified to MIL-I-25017 and listed on the QPL may be used, at the option of the supplier, in JP-4 and JP-5 type fuels.

Currently, concentration requirements for the addition of CI in jet fuel are determined in accordance with the 'Rusting Test Method' specified by MIL-I-25017D. The Rust Test is in keeping with the original purpose of CI to inhibit pipeline and ground system corrosion. However, since 1966 the addition of CI to jet fuels has been mandated primarily for the purpose of lubricity enhancement. The Ball-On-Cylinder Lubricity Evaluator (BOCLE) is recognized as the best available method for providing a relative system of measurement of fuel lubricity. A variety of Ball-On-Cylinder machines, test procedures, test cylinders and reference fluids have been investigated in past years. Recommendations to the Coordinating Research Council (CRC) based on results of an Air Force study completed in August of 1987 (Ref. 5), resulted in standardization of the test apparatus and procedure. With the acceptance of a standard BOCLE test procedure, reevaluation of CI, based on their ability to impart lubricity to jet fuels, was the next step in the Air Force's plan for controlling the lubricity of fuels used in fleet aircraft.

2. QUANTIFICATION OF CORROSION INHIBITORS

Despite dependence on the mandatory use of fuel soluble CI to alleviate lubricity related fuel system wear problems, there is no accepted method for monitoring compliance. Nor is there a means of measuring CI levels at the point of use for detecting additive losses occurring during transportation.

In the past, labor intensive extraction techniques have been proposed for quantitative analysis (Refs. 6 and 7) of CI. These techniques have not been evaluated for broad application to all QPL CI or varying fuel matrices. A simple, direct fuel injection, analytical method to quantify low levels of CI was developed under Task Order No. 6, "Determination of Corrosion Inhibitor in Aviation Fuels" (Ref. 8). This method, using Reverse Phase High Performance Liquid Chromatography (RPHPLC), appears to best satisfy the prerequisites of a reliable, effective, means for determining CI content in jet fuels. The RPHPLC methodology is based on detection of the dilinoleic acid active ingredient found in the most frequently used CI.

Additional refinement of the test method was, however, needed to extend its applicability to all QPL approved CI. A limited survey of QPL CI indicated that most are multi-component mixtures. Increased resolution of CI components was necessary to provide good precision and accuracy for quantification. Further, identification of the specific CI product added to the fuel was believed to be possible, but only if the product components yielded unique chromatograms.

3. PROGRAM OBJECTIVES

The thrust of technical effort described in the following sections was directed at accomplishing the following goals:

- Evaluation of the currently approved QPL CI in terms of lubricity enhancement
- Establishment of minimum effective concentrations for approved CI
- Generation of working curves to profile CI performance in jet fuels
- Development of an approach for incorporating a lubricity requirement into MIL-I-25017 for the purpose of qualifying candidate CI
- Refinement of the RPHPLC method for determining CI content in jet fuels
- Determination of applicability of the RPHPLC method to QPL CI
- Generation of an RPHPLC spectral library of QPL CI in JP-4

SECTION II

EXPERIMENTAL

This investigation focused on two distinct but interrelated efforts. These efforts were conducted simultaneously and together provided an assessment of CI performance and a method to quantify them. The CI evaluations focused on the ability of each of the QPL CI to measurably improve fuel lubricity. The quantification of CI focused on refinement of the recently developed RPHPLC methodology for determining CI content in jet fuels. Applicability of the method to each of the 15 approved QPL CI was also investigated. The following paragraphs discuss test parameters, equipment set up, and experimental approaches used in this investigation.

1. CORROSION INHIBITOR EVALUATIONS

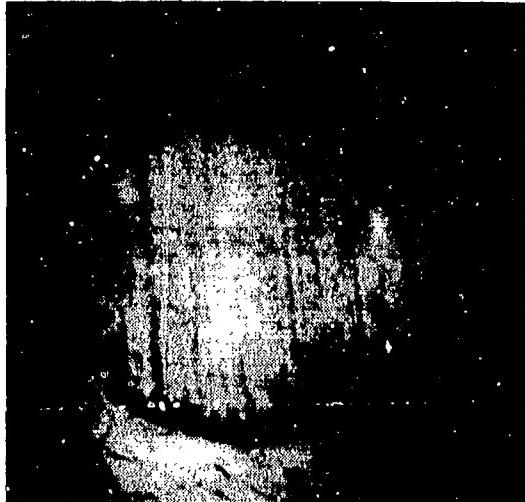
a. Ball-On-Cylinder Lubricity Evaluator (BOCLE)

An InterAv BOCLE was used to assess the ability of each CI to improve the lubricity properties of jet fuels. BOCLE tests were performed according to the standard test procedure approved by the CRC Ball-On-Cylinder Operators' Task Force, "Standard Test Method For Measurement of Lubricity of Liquid Hydrocarbon Fuels By the Ball-On-Cylinder Lubricity Evaluator." The method assesses the boundary lubrication properties of aviation fuels and similar hydrocarbon liquids on rubbing surfaces.

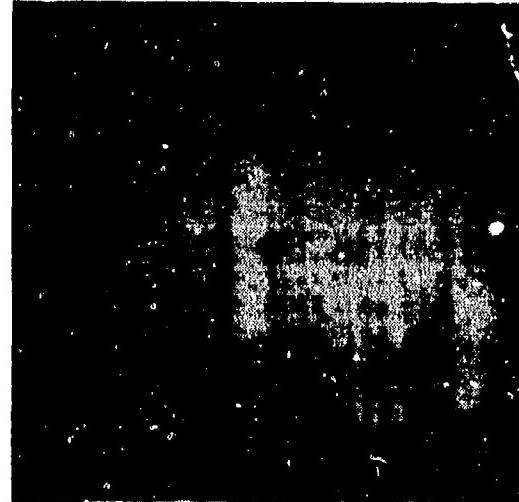
In this method, the test fluid is placed in a reservoir in which the air atmosphere is maintained at 10 percent relative humidity. The fuel temperature during a standard test is maintained at 25°C (77°F). A nonrotating loaded steel ball is held in a vertically mounted chuck and forced against an axially mounted steel test ring. The test ring is rotated at 240 revolutions per minute (rpm) and receives a momentary exposure to the test fluid upon each revolution. At the conclusion of the test, the wear scar generated on the test ball is viewed under a microscope at 100X magnification. A 1-millimeter (mm) graduated reticule permits the major and minor axis of the scar to be measured to the nearest 0.01 mm. The average of the two measurements is reported as the BOCLE wear scar diameter (WSD) and is a measure of the fluid lubricating properties. The smaller the WSD, the better the fuel lubricity. Typical wear scars produced by jet fuels are shown in Figure 1. For the purpose of evaluating lubricity effects at elevated temperatures, an auxiliary Neslab Exacal-100 DD Bath Circulator and a Neslab EN-150 Endocal Flow Through Cooler were interfaced with the existing system.

An overview of the InterAv BOCLE control panel is shown in Figure 2-a. The control panel permits control of test duration, temperature, relative humidity, and ring rotational speed. The base unit is shown in Figure 2-b and is comprised of a fuel reservoir, an axially mounted Falex Ring, a micrometer used for spacing the wear tracks of subsequent runs, and a load beam with test ball installed. The lines shown running to the fuel reservoir provide the means for circulating a fluid medium through the heat exchanger for controlling test temperature.

A reduction in applied load from 1000 to 500 grams represents the only change in test conditions from that described in Draft No. 10 of the CRC BOCLE test procedure. A previous Air Force investigation to standardize the BOCLE test resulted in replacing the AMS 6444 test cylinder with the Falex Ring (Ref. 5). The harder Falex Ring material generated a significantly larger scar than the AMS 6444 cylinder. At a 1000 gram load, the WSD of clay treated fuels sometimes approached or exceeded 1 mm.



JP-4
wear scar



JP-7
wear scar

FD 346595

Figure 1. Typical BOCLE Wear Scars Produced by Jet Fuels

After a series of runs using harsher test fluids, we determined that a 500-gram load was more suitable for maintaining a wear scar within the limits of the 1-mm graduated reticle of the microscope. These tests also indicated that repeatability was enhanced at a 500-gram load. This agreed with past experience that has shown repeatability to be influenced by the size of the wear scar; the larger the wear scar, the more scatter that is typically introduced. Therefore, the BOCLE tests conducted in this program were performed at a 500-gram applied load using the Falex Ring as the standard test specimen. However, subsequent to the completion of this program, a CRC round robin evaluation of the effect of load on test precision was conducted. Statistical analysis of the round robin data found the 1000-gram load to be more reproducible from laboratory to laboratory and, as such, has been incorporated into the proposed test procedure as the standard load.

Figure 3 shows the linear correlation between wear scars produced at 500 and 1000-gram loads. The plot was generated using the average values of 20 laboratories testing nine fuel samples. The best-fit equation of the line is $Y=1.144X-0.006$, where Y equals the WSD at 1000 grams, X equals the WSD at 500 grams, 1.144 is the slope, and -0.006 is the Y intercept. The correlation coefficient is 0.9933, and the standard error of the estimate is 0.015. Both the plot and the equation of the line can be used as a quick reference to relate values generated at 500 grams to 1000 grams.

All Falex Rings used throughout this technical effort were calibrated using two standard reference fluids. These reference fluids are designated as Primary Reference Fluid A and Secondary Reference Fluid B in Draft No. 10 of the CRC BOCLE test procedure. Reference Fluid A is a mixture containing 30 parts per million (ppm) by weight duPont DCI-4A CI/Lubricity Improver in Isopar M. Isopar M is a relatively pure, narrow-cut, isoparaffinic solvent produced by Exxon. The chemical and physical properties of Isopar M are included in Appendix A. Reference Fluid B is 'neat' Isopar M containing no additives.

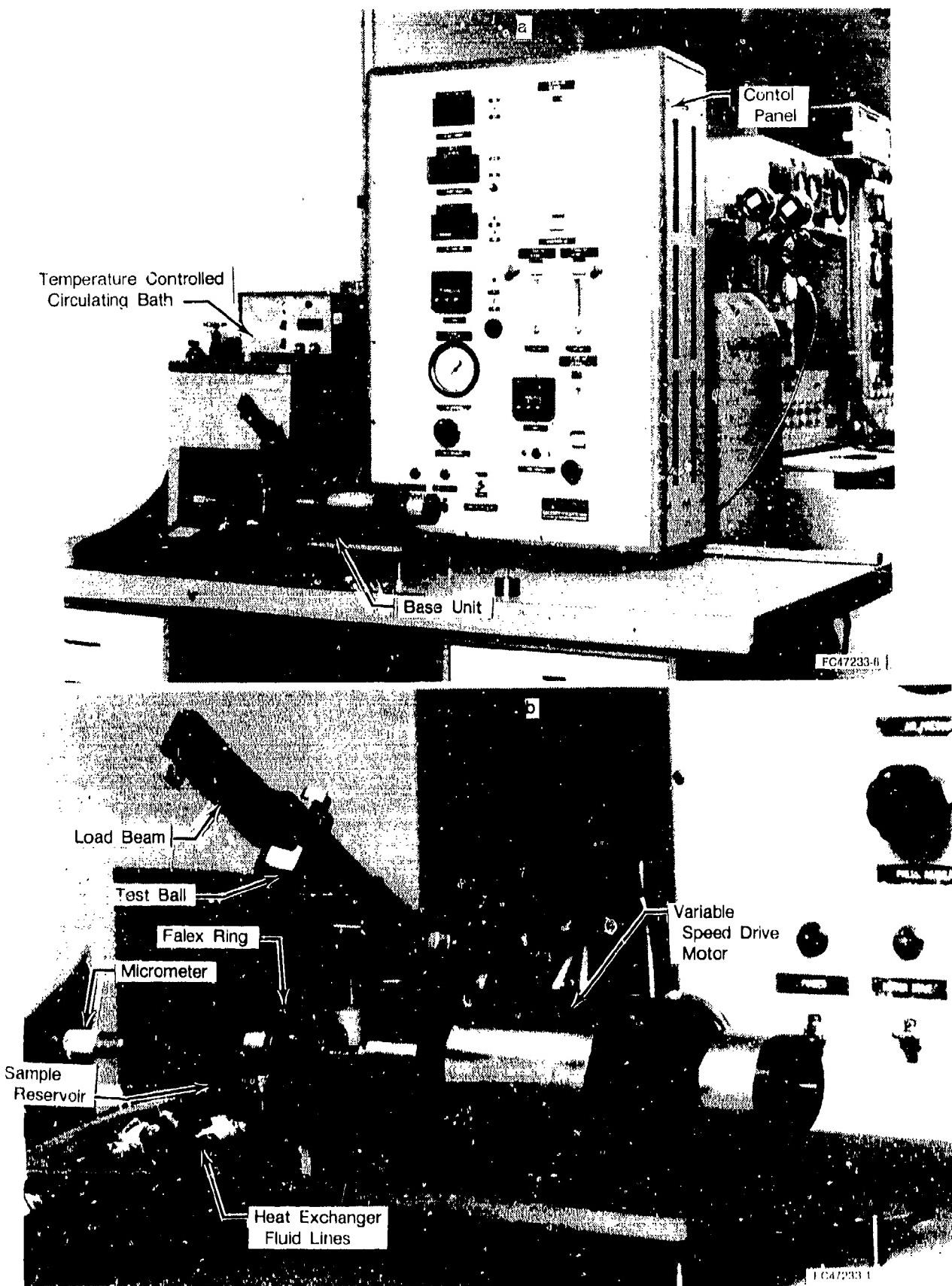


Figure 2. InterAv Ball-On-Cylinder Lubricity Evaluator

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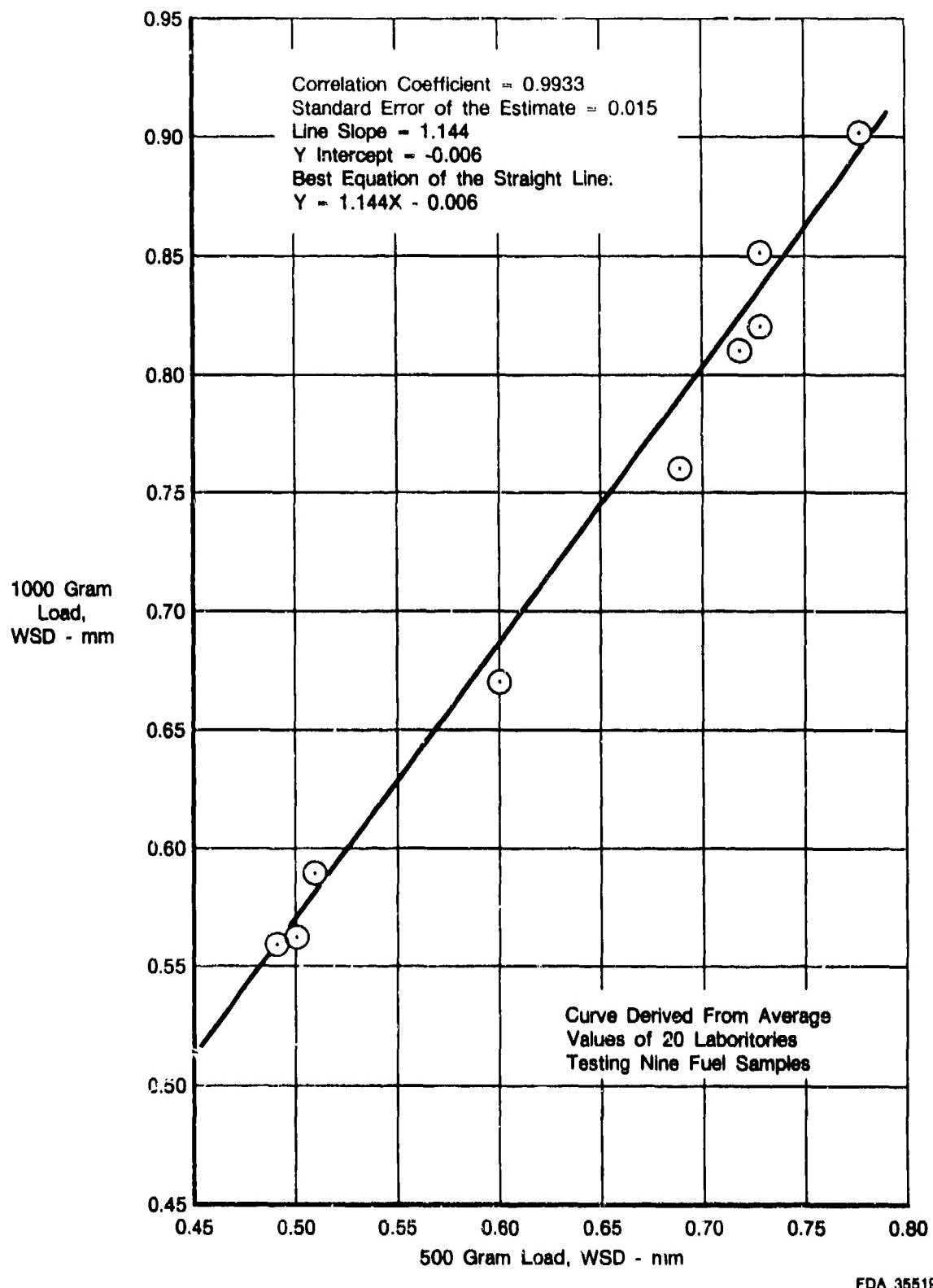


Figure 3. BOCLE Load Correlation

Reference fluid A was used specifically to qualify the test rings for use in BOCLE testing. A WSD of 0.50 ± 0.02 mm was used as the qualifying criteria. Reference Fluid B (neat Isopar M) is a considerably harsher fluid than that of the Isopar M/DCI-4A mixture and as a result produces a significantly larger wear scar. It is, therefore, sensitive to ring contamination and so was used to ensure that proper cleaning precautions had been followed.

In addition to qualifying the test rings using the primary and secondary reference fluids, conformance to material specifications for surface finish and hardness was verified. These measurements also permitted the relationship between the specified material properties and the accepted reference fluid calibration value to be established. Verification of the 20- to 30-microinch (μ) surface finish was accomplished using a Sheffield Model E-20A Surface Texture Measuring Instrument. Rockwell hardness was verified using a Wilson Mechanical Rockwell Superficial Hardness Tester.

b. Additives Evaluated

BOCLE tests were performed on each of the CI/Lubricity Improvers approved for use by the MIL-I-25017D QPL. The CI evaluated, manufacturer's designation, and specified relative effective, minimum effective, and maximum allowable concentrations are listed in QPL-25017-15 that is contained in Appendix B. During the course of the investigation, the most recent revision of the QPL, QPL-25017-15 issued in January 1987, resulted in the deletion of P-3305 (Unichema Chemie B.V., Netherlands) from the QPL. Two new CI were added to the revised QPL: Nucem PCI-105 and Welchem 91120. Both the former and the latter CI were included in the investigation. Fresh samples of CI were procured from the manufacturers.

c. Test Fuels

CI performance was evaluated in four test fuels: neat Isopar M, JP-4, JP-8, and JP-5. The matrix fuels were stored in 55-gallon epoxy-lined drums. With the exception of Isopar M, 20 gallons of each fuel type were stripped of additives, naturally occurring lubricity enhancers (polar compounds), and contaminants by slow percolation through oven dried Attapulgus clay. Clay treating (CT) was performed according to Annex A of ASTM D2550. The degree of fluid harshness attained was verified by performing BOCLE tests upon completion of the clay treating. In instances where a BOCLE WSD of greater than 0.75 mm was not obtained, the fuel was subjected to additional passes through the clay. The harshness of the matrix fuels was verified a second time prior to preparation of each set of CI/fuel blends.

d. Additive/Fuel Blends

To determine effective CI concentrations in each of the four test fuels, testing was performed at nine concentrations ranging from zero to the maximum allowable concentration permitted for each CI in QPL-25017-15. Additive/fuel blends were formulated from a 100-ppm concentrate at levels of 0, 1.5, 3, 6, 9, 12, 15, 20, and 30 ppm by weight. This range was extended for two of the CI having maximum allowable concentrations of 42 ppm. To provide a fair assessment of the effective concentration of the CI in each of the four different test fuels, the CI were blended at the various concentrations in ppm by weight rather than in grams per cubic meter (g/m^3). Depending on the density of the fuel, a maximum allowable concentration of $22.5 \text{ g}/\text{m}^3$ ranges from approximately 26 to 30 ppm.

e. Test Temperatures

BOCLE WSD as a function of CI concentration were generated at 25°C (77°F). In three of the four test fuels, CI were also evaluated at 75°C (167°F) to assess the effect of temperature on additive performance.

f. Summary of Test Matrix

A summary of the test materials, CI concentrations, and test temperatures is shown below:

BOCLE:	Falex Ring, 500g applied load
QPL CI/Lubricity Improvers:	16
CI Levels Evaluated (ppm):	0, 1.5, 3, 6, 9, 12, 15, 20, 30, 42
Test Fuels:	Neat Isopar M, CT JP-4, CT JP-8, CT JP-5
Test Temperatures:	25°C (77°F), 75°C (167°F)

2. REFINEMENT OF RPHPLC METHOD FOR DETERMINING CI CONTENT IN JET FUELS

a. Equipment and Instrumental Conditions

Based on previous research reported in Reference 6, the following equipment and instrumental conditions were used to begin the method optimization:

A Varian Model 5560 Ternary Liquid Chromatograph as shown in Figure 4 was used to perform all HPLC analyses in this study. It was equipped with a Varian Model UV200 variable wavelength ultraviolet-visible detector set to 202 nanometer (nm) with a 0.5-second response time, a Rheodyne Model 7125 injector valve with 50 microliter (μL) sample loop, and an electronic column heater. Quantification was accomplished using a Varian Model Vista 402 Chromatography Data System. Baseline treatment was performed automatically by the data system software. All calibrations were performed in the External Standard mode.



FC 46315

Figure 4. Varian High Performance Liquid Chromatograph

The following HPLC bonded phase columns were evaluated: cyano (Alltech Associates, Deerfield, IL); amino, cyano and phenyl (Brownlee Labs, Santa Clara, CA); carboxymethyl weak ion exchange (Toyo Sota, Japan); phenyl sulfonic acid strong cation exchange and quaternary amine strong anion exchange (Whatman, obtained from Alltech Associates).

The mobile phase compositions evaluated included blends of methanol, isopropanol, and aqueous buffers.

Samples and standards were injected into the chromatograph, without any pretreatment, via a 50- μ L sample loop. After the additive compounds eluted, a column wash and reequilibration program was necessary to remove the residual fuel sample. The ternary (3 solvent) capability of the chromatograph was used to perform this function.

Standards were prepared in 125-milliliter (μ L) Teflon bottles (Nalge Co., Rochester, NY).

b. Materials

- HPLC grade methanol, isopropanol and water were obtained from Burdick and Jackson (Muskegon, MI).
- Potassium phosphate, monobasic (KH_2PO_4) and sodium hydroxide (NaOH) were ACS Analytical Reagent grade and were obtained from Mallinckrodt, Inc. (Paris, KY).
- EMPOL 1010, a 97 percent pure dilinoleic acid, and EMPOL 1041, an 80 percent trilinoleic acid (20 percent dimer), were obtained from Emery Chemical Company (Cincinnati, OH).
- Purified linoleic acid was obtained from Fisher Scientific Company, (Fair Lawn, NJ).
- The 15 CI products listed in the QPL-25017 -15 were obtained from the respective suppliers.
- Clay treated JP-4 fuel was prepared by filtering JP-4 through a glass column packed with Attapulgus clay per ASTM D2550.
- Additive free JP-4 was obtained from Sun Oil Company (Philadelphia, PA).

SECTION III

RESULTS AND DISCUSSION

The results and significance of the BOCLE tests performed on 16 corrosion inhibitors are discussed in the paragraphs that follow. Results and discussion of work directed at refining the RPHPLC method for determining CI content in jet fuels and applicability of the methodology to CI approved under MIL-I-25017D are also presented.

1. CORROSION INHIBITOR EVALUATIONS

a. Lubricity Test Data

Tables of the BOCLE data generated for each of the CI in each test fuel, over the range of concentrations at which they were tested, are included in Appendix C. Profiles of additive performance were generated for each CI in the form of polynomial curve fits plotted from the test data. These plots provide a means for determining the amount of additive required to provide sufficient lubricity properties to jet fuels. The plots also provide an avenue for tracking deterioration in fuel lubricity as levels of CI are depleted. These curve fits are presented in Appendix D, indexed by fuel type.

b. Criteria Used to Assess Performance

In order to assess the effectiveness of the CI, guidelines and criteria were established to permit comparison of the responsiveness of one CI to another. Among the criteria considered was the amount of additive required to provide relative effective lubricity enhancement. For the purpose of this discussion, the terminology 'relative effective concentration' is defined as the concentration of CI required to provide a BOCLE WSD equal to 0.60 mm. The 0.60 mm WSD is currently under consideration by the Air Force to describe minimum acceptable lubricity. This value was selected on the basis of an Air Force survey of hydraulic fuel pumps that had experienced problems when exposed to fuel having a WSD of greater than 0.60 mm. In rating CI performance, consideration was also given to the lubricity improvement that was attainable at maximum allowable concentration. Twenty-two and a half grams per cubic meter was the maximum allowable concentration permitted by QPL-25017 for all but two of the CI evaluated. The two exceptions are Unichema P-3305 (excluded from the QPL in the January 1987 revision) and Tolad 245. Both are permitted a maximum allowable concentration of 42 ppm (31.5 g/m^3).

Therefore, examination of the BOCLE results considered (1) 'relative effective concentration', defined as the level of CI required to reduce the BOCLE WSD to 0.60 mm (2) 'maximum effective concentration', defined as that concentration at which no further reduction in WSD is apparent with continued increases in CI concentration; and (3) maximum lubricity achieved, i.e., WSD at the maximum allowable concentration.

c. Performance Profiles

Computer software was developed to permit archiving and plotting of the BOCLE test data. Second, third, and fourth order polynomial curve fits best-fit the test data for plotting WSD as a function of concentration. The curve fits were used as a tool to establish relative effective concentrations for each CI and for determining the effect of fuel type and temperature on CI response. Figure 4 is an example of the curves produced by the software. For quick reference, a dashed line was drawn across the plot by the computer to represent the targeted 0.60 mm WSD lubricity value. The point at which the plotted curve crossed the dashed line was calculated from the polynomial equation and printed out on the plot as the "relative effective concentration." As

shown in Figure 5, the concentration requirements per QPL-25017 the accuracy of the curve fit data (standard error of the estimate (SEE)), and correlation coefficient (R) are also documented on the plot. A complete set of curve fits, profiling additive performance for each QPL CI, are contained in Appendix D, indexed by fuel type.

While interpreting the curves shown for each CI, consideration should be given to small variations in additive response that may appear unduly emphasized by the graph. In reality, the data fall within the established limits of test repeatability (0.03 mm). Although varying in profile, the curves for each CI assessed at 25°C (77°F) responded predictably to increasing CI concentrations. At maximum allowable concentration, WSD ranged from 0.47 to 0.61 mm for the 16 CI tested in the four fuel types. In at least three out of the four test fuels, all but four of the 16 CI had achieved maximum effectiveness upon reaching maximum allowable concentration. As shown by the plots in Appendix D, the majority of CI exhibited a plateau between 20 and 30 ppm, showing no further reduction in WSD with increased concentration. Six of the CI, however, did show evidence of a continued reduction in WSD with increasing concentration.

Curve fits comparing IPC-4410 and Tolad 249 in CT JP-4 are shown in Figures 5 and 6, respectively. IPC-4410, as shown in Figure 5, exhibits outstanding lubricity improving properties with a 0.48 mm WSD achieved at maximum allowable concentration. Additive efficiency of IPC-4410 in JP-4 is reflected by the small amount of CI required to meet the required 0.60 mm WSD. IPC-4410 performed equally well in CT JP-8 and CT JP-5. As shown by the curve in Figure 6, Tolad 249 failed to meet the 0.60-mm WSD criteria for lubricity enhancement even at its maximum allowable concentration. Field experience supports the test data, indicating that Tolad 249 is among the least effective CI currently on the approved QPL.

d. Effect of Fuel Type on Additive Performance

It was suspected during the early stages of this investigation that some additives would respond differently to different type fuels. In general, fuel type had little effect on additive performance. These were, however, clay treated fuels. It is possible that unique responses to additives could occur with fuels containing different polar compounds. In the clay treated samples, the BOCLE WSD at maximum allowable concentration, and the level of CI required to achieve a 0.60-mm WSD, were relatively consistent from fuel type to fuel type. Some variation, however, was observed. As shown in Table 1, the most significant variation was that of P-3305 (no longer an approved CI) in JP-5. This is apparent when comparing its performance in JP-5 to the other three test fuels. It was thought that the slight differences in the performance of an additive from one fuel type to another could be attributed to one, or a combination, of the following causes:

- CI depletion
- Unique CI response to the properties of a specific fuel type
- Small variations in CI active ingredient between stock solutions caused by vaporization of diluent during weighing/blending procedure
- Variations in volatility of the fuels that may cause concentration of CI during the BOCLE test
- Unknown test variable(s)

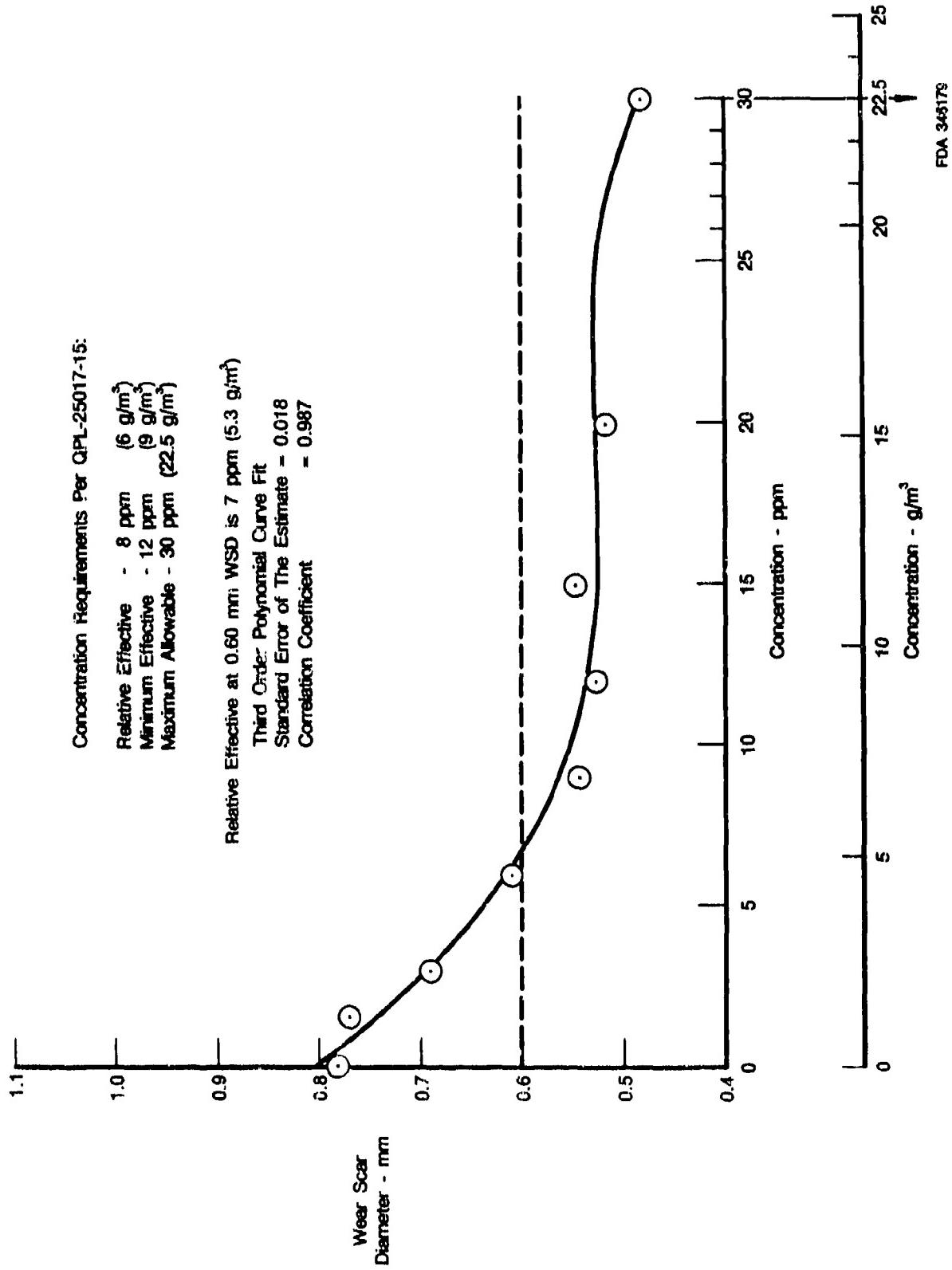


Figure 5. Effect of IPC-4410 in Clay Treated JP-4

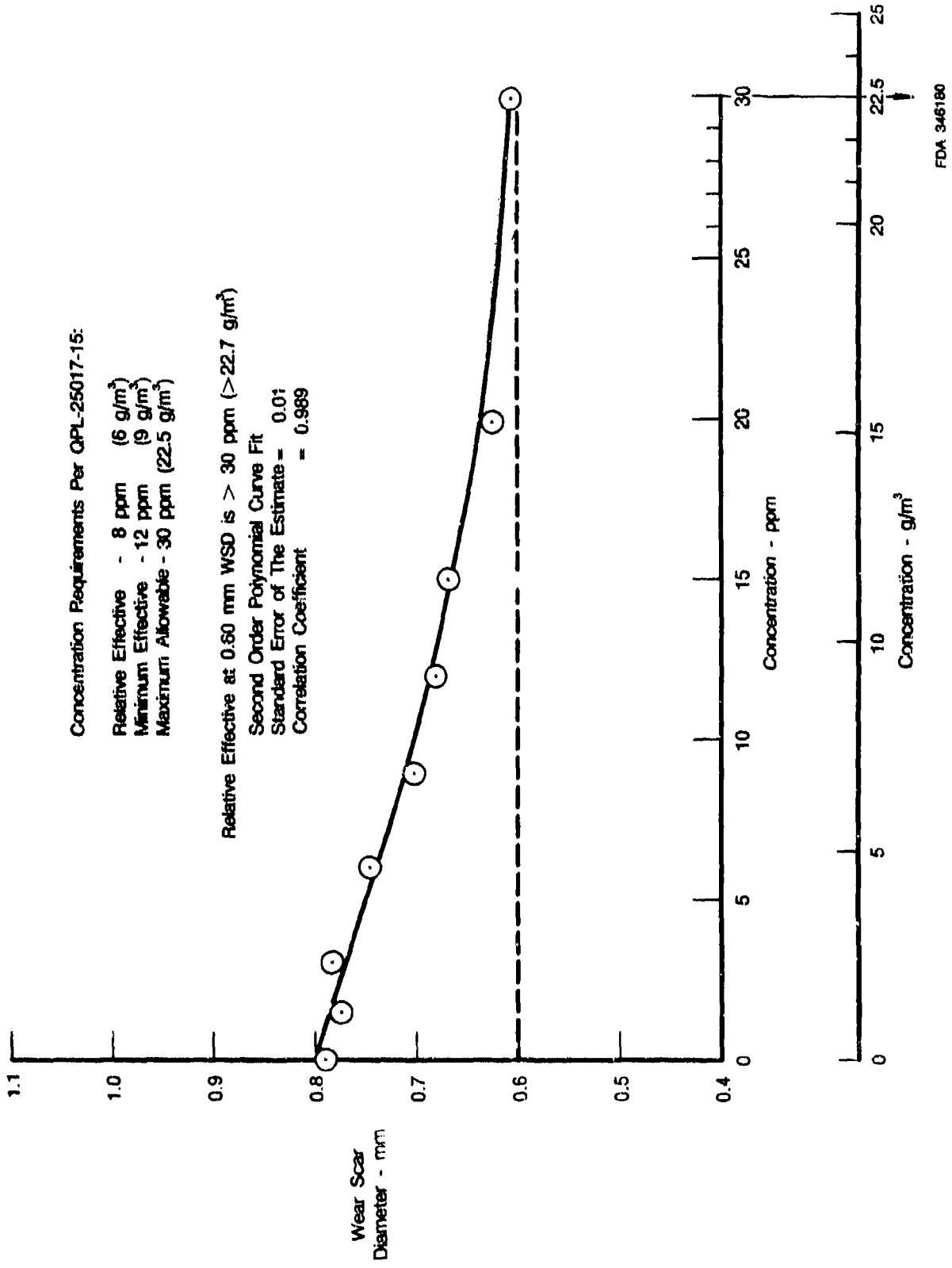


Figure 6. Effect of Toldad 249 in Clay Treated JP-4

TABLE 1. Effective Corrosion Inhibitor Concentrations for Lubricity Improvement

Corrosion Inhibitor	Concentration Required to Achieve 0.60 mm BOCLE WSD g/m ³						BOCLE WSD at Maximum Allowable Concentration mm						All Fuel Types	
	Isopar M	CT JP-4	CT JP-5	CT JP-8	Average Range	Average Concentration Required	Isopar M	CT JP-4	CT JP-5	CT JP-8	Average Range	Average WSD		
IPC-4410	8.3	5.3	6.5	6.4	3.0	6.6	0.51	0.48	0.48	0.48	0.03	0.49		
NALCO 5405	9.8	6.8	8.1	7.9	3.0	8.2	0.48	0.50	0.52	0.48	0.04	0.50		
ARCO IPC-4445	14.4	9.0	11.3	11.9	5.4	11.6	0.54	0.50	0.56	0.54	0.08	0.54		
UNICHEM P-3305	7.6	8.3	18.9	9.5	11.3	11.1	0.47	0.52	0.51	0.52	0.06	0.50		
NALCO 5403	10.6	9.0	8.9	9.5	1.7	9.5	0.53	0.53	0.50	0.50	0.03	0.52		
UNICOR J	6.8	6.8	8.1	8.7	1.9	7.6	0.47	0.53	0.48	0.54	0.08	0.50		
MOBILAD F-800	8.3	6.8	6.5	9.5	2.7	7.8	0.49	0.52	0.50	0.48	0.04	0.50		
TOLAD 245	20.4	21.1	19.4	19.1	2.0	20.0	0.55	0.54	0.54	0.55	0.01	0.54		
DCI-6A	9.1	8.3	6.5	8.7	2.6	8.2	0.48	0.54	0.48	0.52	0.06	0.50		
LUBRIZOL 541	13.6	13.6	10.5	11.9	3.1	12.4	0.54	0.54	0.58	0.55	0.02	0.55		
DCI-4A	6.8	9.0	6.5	7.2	2.5	7.4	0.48	0.55	0.48	0.50	0.07	0.50		
HITEC E-580	12.1	8.3	11.3	11.1	3.8	10.7	0.52	0.55	0.51	0.54	0.04	0.53		
APOLLO PRI-19	15.1	14.3	13.0	14.3	2.1	14.2	0.54	0.56	0.52	0.57	0.05	0.55		
TOLAD 249	21.9	>22.6	23.5	23.1	1.6	>22.8*	0.59	0.61	0.60	0.60	0.02	0.60		
WELCHEM 91120	12.1	8.3	11.3	12.7	4.4	11.1	0.56	0.52	0.54	0.54	0.04	0.54		
NUCHEM PCI-105	13.6	12.8	13.8	11.9	1.9	13.0	0.55	0.54	0.54	0.52	0.03	0.54		

*Exceeds Maximum Allowable Concentration per QPL-25017-15

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A brief study was conducted to investigate the effect of CI depletion on the test data. The effect of CI depletion on BOCLE results was considered in relation to the time between preparation of the 100 ppm concentrate and preparation of the subsequent blends. Also investigated was the effect of time between preparation of the individual blends and performance of the BOCLE tests. A series of CI profiles previously generated were repeated. BOCLE tests on the individual blends were performed within 24 hours of preparation of the initial 100 ppm concentrate. RPHPLC was used to monitor CI depletion rates. No evidence was found that the BOCLE results had been affected by depletion of CI or that significant plating out of additive had occurred during the 24-hour period. The newly generated curves were consistent with those previously reported for the other three test fuels.

e. Effective Concentrations for Lubricity Improvement

The effective concentrations for lubricity improvement, based on the 0.60 WSD criteria, are shown in Table 1 for each of the 16 CI in each of the four test fuels. Military specifications require the addition of CI to JP-4, JP-5, and JP-8. It is unlikely that a future revision to the military specification will call out varying relative, minimum, and maximum concentrations based on fuel type alone. Fuel type has been shown to have little effect on additive performance. Since there is a need to know how an additive will respond in general to any current JP fuel, much of the following discussion will focus on the average test results for all matrix fuels cumulatively as opposed to addressing each fuel type individually. Table 1 permits independent comparison of test fuels.

In addition to data for each specific fuel, the average concentration required in all test fuels to achieve minimum lubricity improvement is shown in Table 1. Only six of the CI evaluated exhibited a 0.60 mm WSD at concentration levels of 9 g/m³ or less. Nine g/m³ is the 'minimum effective concentration' defined by QPL-25017-15, while 0.60 mm WSD is the Air Force proposed

value for determining minimum acceptable lubricity improvement. Of the remaining ten CI, eight met the 0.60-mm WSD criteria between 10 and 14 g/m³, one approached its maximum allowable concentration, and one CI (Tolad 249) exceeded maximum allowable concentration. Looking at the test fuels independently, only 5 CI in Isopar M, 5 in JP-8, 7 in JP-5, and 11 in JP-4 met the desired 0.60-mm WSD criteria for minimum effective lubricity improvement at the minimum effective concentration of 9 g/m³ designated by QPL-25017-15. The improved performance of many CI in JP-4 may be a result of evaporation of light ends during the BOCLE test, thereby concentrating CI. Maximum effective concentration (that in which no further reduction in WSD was realized) ranged from 9 to 31.5 g/m³.

f. Performance Ranking

Based on their performance as lubricity improvers, the 15 currently approved CI fell into three distinct groups. Grouping of CI performance was based on the average results of all test fuels. Each performance group, as shown in Table 2, was distinctive in relation to both total reduction in WSD achieved at maximum allowable concentration as well as the concentration required to achieve 0.60 mm WSD.

Group No. 1 consisted of six CI in which similar maximum lubricity improvement of 0.49 to 0.50 mm WSD (essentially the same) was achieved. Concentrations required to achieve a 0.60 mm WSD ranged from 6.6 to 8.2 g/m³. This concentration range conformed to the 9 g/m³ minimum effective concentration requirement set forth by QPL-25017-15. The CI in Group No. 1 were extremely efficient in low level response to providing and maintaining excellent lubricity enhancement.

Group No. 2 consisted of eight CI providing a maximum lubricity improvement of 0.52 to 0.55 mm WSD. Concentrations required to achieve a 0.60 mm WSD ranged from 9.5 to 12.4 g/m³. With the exception of one CI, this group exceeded the QPL defined minimum effective concentration (Tolad 245 has a 22.5 g/m³ minimum effective concentration while the remaining CI have requirements of 9 g/m³). This group of CI exhibited good to fair lubricity enhancing properties.

Group No. 3 consisted of one CI (Tolad 249) that provided an average maximum lubricity improvement of 0.60 mm WSD. In three out of four test fuels, maximum allowable concentration was exceeded prior to achieving this value. Tolad 249 was shown to be the least effective CI in providing adequate lubricity enhancement of jet fuels.

TABLE 2.
GROUPING OF CORROSION INHIBITOR PERFORMANCE

Corrosion Inhibitor	Average, All Test Fuels	
	WSD at MAC mm	Concentration Required To Achieve 0.60 mm WSD g/m ³
<i>Group No. 1</i>		
IPC 4410	0.49	6.8
NALCO 5405	0.50	8.2
UNICOR J	0.50	7.6
MOBILAD-F800	0.50	7.8
DCI-6A	0.50	8.2
DCI-4A	0.50	7.4
<i>Group No. 2</i>		
NALCO 5403	0.52	9.5
HITEC E-580	0.53	10.7
IPC 4445	0.54	11.6
TOLAD 245	0.54	20.0
WELCHEM 91120	0.54	11.1
NUCHEM PCI-105	0.54	13.0
APOLLO PRI-19	0.55	14.2
LUBRIZOL 541	0.55	12.4
<i>Group No. 3</i>		
TOLAD 249	0.60	>22.8
Note:		
1. QPL --- MIL-I-25017D Qualified Products List		
2. Average WSD-Average Wear Scar Diameter of CI in Four Test Fuels		
3. MAC --- Maximum Allowable Concentration Per QPL-25017-15		
4. QPL Minimum Effective Concentration 9 g/m ³ , Except TOLAD 245 22.5 g/m ³		

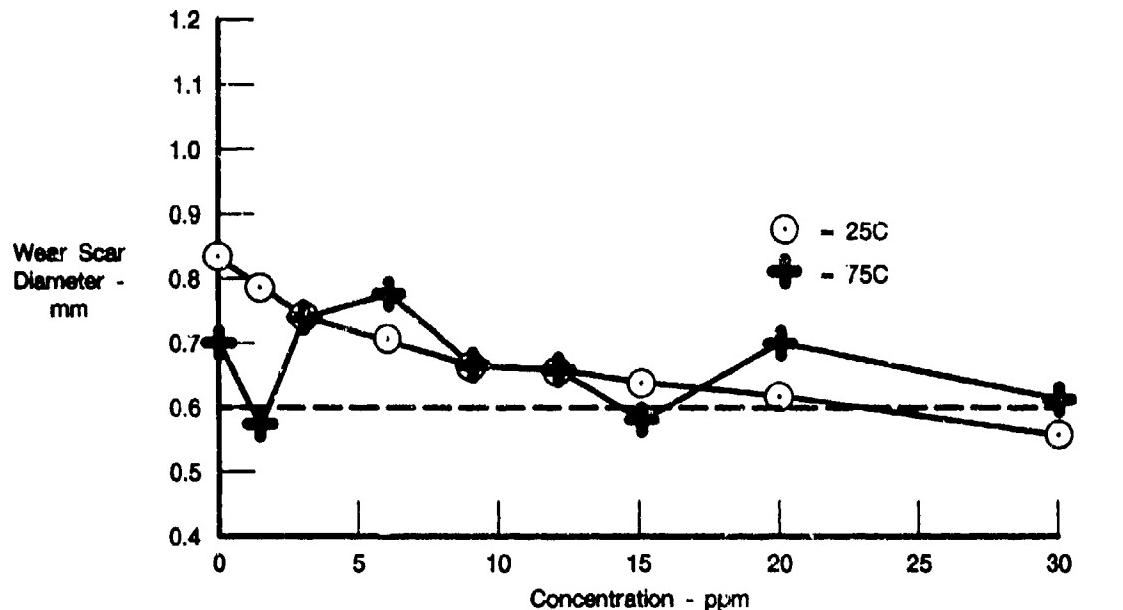
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While the grouping of CI performance is subjective, the thought process in discerning between the three groups takes into consideration not only the total reduction in WSD but also the military specification requirements for minimum effective concentration. Of those additives evaluated, IPC-4410 was among the most effective while Tolad 249 was shown to be the least effective.

g. Effect of Temperature

BOCLE tests at 75°C (167°F) were performed on the CI/fuel blends to assess the effect of temperature on CI performance. As predicted, wear scars generated at 75°C (167°F) were in most cases measurably larger than those produced for the same fuel blends at 25°C (77°F). This supported earlier work by the Naval Air Propulsion Center (NAPC) (Ref. 8) and P&W (Ref. 10) indicating that temperature has a dramatic effect on fuel lubricity. Plots generated from the 75°C (167°F) runs, however, were shown to be extremely erratic. The predictable curves exhibited by the 25°C (77°F) data, showing enhanced lubricity as a function of concentration, were not apparent when testing at 75°C (167°F). Data scatter and lack of repeatability made valid interpretation of the test results difficult, if not impossible, at 75°C (167°F).

Figures 7 and 8 are representative of the phenomenon observed during the elevated temperature tests. To verify that the erratic data was the result of random, temperature-induced scatter, the entire series of BOCLE runs were repeated from 0 to 30 ppm at 75°C (167°F) for two CI. The resulting profiles were significantly different from the original plots. Both CIs continued to exhibit nonrepeating, random data scatter. It was concluded that the validity and usefulness of the data was questionable. With the concurrence of the Air Force Project Engineer, BOCLE testing at elevated temperature was terminated.

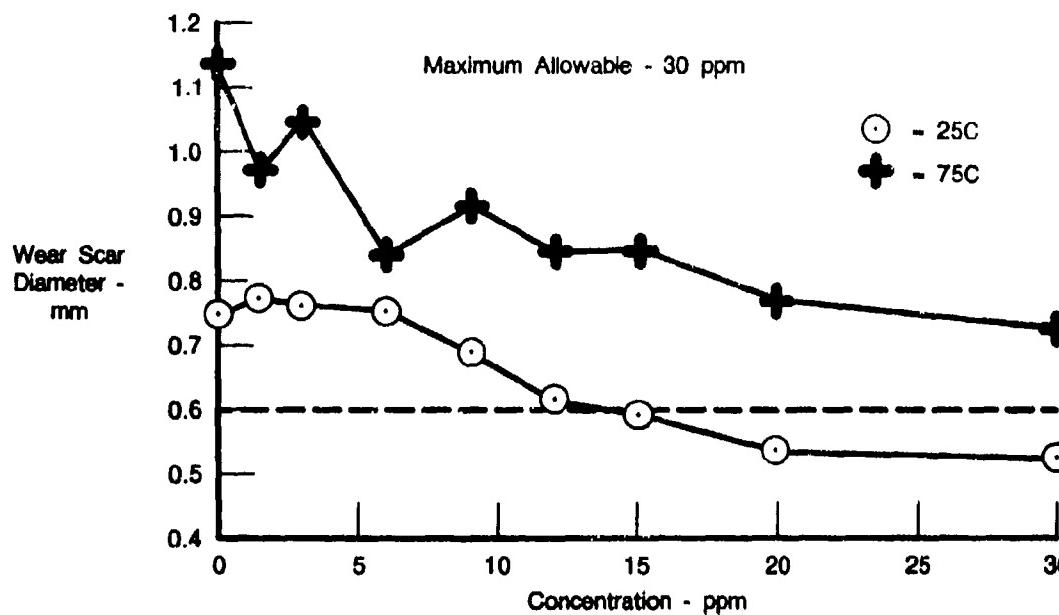


FDA 346195

Figure 7. — Effect of Temperature on PRI-19 in Clay Treated JP-4

Temperature has an effect on the way CI function in different wear modes. Although not fully understood, it is suspected that more than one type of wear occurs during BOCLE operation. Wear mechanisms that occur in the boundary lubrication regime that could be applicable to BOCLE operation include (1) corrosive wear, (2) abrasive wear, and (3) adhesive wear.

Corrosive wear occurs when oxygen reacts with metal surfaces to form metal oxides. These oxides are easily worn away providing fresh surfaces for further oxidation. CI function by adsorption of the CI polar carboxy group to the metal surface. Thus forming a molecular boundary layer that acts as a barrier to oxygen and moisture. Adhesive and abrasive wear occurs when asperities of two metal surfaces come into contact. CI function to provide a molecular boundary layer between the two surfaces. In abrasive or adhesive wear, asperity contact area can grow due to the high normal and tangential stresses on the metal so that the trapped boundary film may be stretched until it ruptures. Local heating can weaken the adsorption forces of the surface film. Simple polar compounds desorb under high temperatures.



FDA 346196

Figure 8. — Effect of Temperature on Hitec E-580 in Isopar M

It is suspected that the data scatter observed at elevated temperature may be a result of opposing effects of the following: increased wear rates, desorption of CI, and increased fuel oxidation rates. Temperature increases the rate of corrosive wear by accelerating the rate of corrosion reactions at the metal surface. In addition, temperature has been shown to increase the rates of adhesive and abrasive wear. The latter has been demonstrated by the increase in wear observed in an inert environment. An increase in temperature will also increase the rate of fuel oxidation reactions that has been shown to reduce wear rates. Fuel decomposition during BOCLE testing is evident from the brownish black residue observed on the test ball at the point of contact. Consequently, in any given test, there may be an ongoing competition between increased wear rates caused by higher temperatures and reduced wear rates due to fuel oxidation.

While not clearly understood, temperature does appear to have a significant effect on CI performance. However, it is apparent that under standard operating conditions, assessment of the effects of temperature on fuel lubricity is beyond the capabilities of the BOCLE.

2. PROPOSED MIL-I-25017 LUBRICITY REQUIREMENT

A thorough review of MIL-I-25017D, which is included in Appendix E, was performed and the historical background of CIs researched. Current Air Force quality assurance and fleet support needs were compared to those of the past. The review sought to determine how the current CI specification could be modified to address a product's ability to improve fuel lubricity, as well as inhibit pipeline and ground fuel system corrosion. We found that a requirement for lubricity enhancement could be easily incorporated into the current specification with no significant effect on other criteria used in qualifying a candidate CI.

There are 12 tests listed in the military specification used to qualify CIs. These tests are directed at controlling CI properties, minimizing the effect on fuel properties, and in establishing

relative effective (REC), minimum effective (MEC), and maximum allowable (MAC) concentrations.

The twelve test criteria consist of the following:

- 1) Solubility — At MAC, there can be no precipitation, cloudiness or other evidence of insolubility.
- 2) Compatibility — At MAC, the CI must be compatible with all other CI currently qualified and with approved static dissipator additives.
- 3) Rust Test — Establishes REC, the lowest concentration yielding a passing result.
- 4) Water Separation Index, Modified (WSIM) — One of two primary criteria used in establishing MAC: the highest concentration giving a WSIM value of 70 or higher.
- 5) Electrical Conductivity — The second of two primary criteria used in establishing MAC: the highest concentration giving less than a 40-percent change in electrical conductivity with fuel containing static dissipator additive.
- 6) Ash Content — Shall not exceed 0.10 percent when determined in accordance with ASTM D482.
- 7) Pour Point — Shall not exceed — 18°C when determined in accordance with ASTM D97.
- 8) Storage Stability — Shall show no evidence of gross separation or degradation after storage for 12 months.
- 9) Induction System Deposits — Applicable if CI to be qualified for motor gasolines.
- 10) Emulsification Tendency — Applicable if CI to be qualified for use in motor gasolines and diesel fuels.
- 11) Accelerated Stability — Applicable if CI to be qualified for use in diesel fuels. Determines formation of total insolubles in accordance with ASTM D2274 at MAC.
- 12) Engine Test — Must pass 100-hour engine test using JP-4 containing CI at 2 times the MAC. Shall indicate no excessive deposits, wear or corrosion attributed to the inhibitor.

Currently, CI effectiveness is evaluated in terms of corrosion inhibition and is assessed primarily on passing the rust test. The lowest concentration at which a CI passes the rust test is defined as the REC, and cannot be less than 6 g/m³. The "not less than 6 g/m³" requirement originates from early development work in which the rust test was shown to exhibit poor precision at low concentrations. The range of concentrations permissible for use in fuels is derived in part from the REC. MEC is specified as 1.5 times REC and cannot be less than 9 g/m³.

(1.5 times 6 g/m³). MAC is governed by a number of considerations; the most significant are WSIM and electrical conductivity. MIL-I-25017D defines MAC as the lowest of the following:

- Fifty-four grams of inhibitor per cubic meter of fuel.
- Four times the REC.
- The highest concentration giving a WSIM value of 70 or greater.
- The highest concentration giving less than a 40-percent change in electrical conductivity with fuel containing static dissipator.

MIL-I-25017D also specifies that the MAC shall be equal to, or greater than, the MEC and shall be a value evenly divisible by 4.5 within a range of 9 to 54 g/m³.

Minimal changes to the current specification would be necessary to incorporate lubricity enhancement as an additional criteria for qualifying CI. Inclusion of a lubricity requirement can be most readily accomplished by redefining REC while leaving the requirements for MEC and MAC unchanged. Maintaining the MEC as 1.5 times the REC is recommended to compensate for (1) blending errors at the refinery, (2) losses during transport and storage, and (3) variations in performance between fuels. MAC would continue to define upper limits. In this way, the basic test requirements are unaffected by the modification. Redefining REC would entail incorporating a 0.60-mm BOCLE WSD lubricity requirement along with the rust test, as well as the following stipulations: (1) REC not exceed 36 g/m³ (if it did, then MAC would exceed the 54 g/m³ limit); (2) 1.5 times the REC not yield a WSIM value less than 70; and (3) 1.5 times the REC not yield greater than 40 percent change in electrical conductivity with fuel containing static dissipator additive.

The latter three stipulations act as a cutoff to prevent those CI that require large concentrations to meet a 0.60-mm BOCLE WSD from exceeding the current MAC requirements. Inclusion of the above restrictions would also permit a step-wise progression for qualifying a CI; eliminating the need for further testing of CI that did not meet the WSIM and electrical conductivity values ultimately required of them at MAC.

The following proposes a rewording of the current MIL-I-25017D specification based on the above discussion. Additional requirements for defining REC and those that have been modified for the current specification are denoted by an asterisk (*).

RELATIVE EFFECTIVE CONCENTRATION (SEC 3.5)

- 1) *Shall be defined as the lowest concentration giving both a passing result in the rust test (Sec 4.6.3) and a maximum BOCLE WSD of 0.60 mm.
- 2) The REC shall not be less than 6 g/m³.
- 3) *The REC shall not exceed 36 g/m³.
- 4) *One and a half times the REC shall not yield a WSIM value less than 70.
- 5) *One and a half times the REC shall not yield greater than 40 percent change in electrical conductivity with fuel containing static dissipator additive.

MINIMUM EFFECTIVE CONCENTRATION (SEC 3.6)

- 1) Shall be defined as 1.5 times the REC.
- 2) Shall not be less than 9 g/m³.
- 3) *Shall not exceed MAC.

MAXIMUM ALLOWABLE CONCENTRATION (SEC 3.7)

The MAC shall be equal to, or greater than, the MEC and shall be the lowest of the following:

- 1) Fifty-four g/m³.
- 2) Four times the REC.
- 3) The highest concentration giving a WSIM value of 70 or higher.
- 4) The highest concentration giving less than 40 percent change in electrical conductivity with fuel containing static dissipator additive.

The effect of redefining relative effective concentration, as shown in Table 3, indicates that MEC would be increased for all but two of the currently approved CI. Three CI would be disqualified based on the above revisions. Those CI failing to meet the new requirements are Tolad 249, Lubrizol 541, and Nuchem PCI 105.

Tolad 249 would fail to qualify on the basis of its limited lubricity improving properties. In determining its REC by the new guidelines, Tolad 249 was unable to achieve a BOCLE WSD of less than or equal to 0.60 mm at concentrations up to 22.5 g/m³. Since its MAC is 22.5 g/m³, Tolad 249 would be disqualified.

Although Lubrizol 541 meets the lubricity requirement at a REC of 13.6 g/m³, its MEC ($1.5 \times 13.6 \text{ g/m}^3 = 20.4 \text{ g/m}^3$) exceeds its current MAC, and as a result would fail to qualify. The same is true for Nuchem PCI-105, whose redefined REC exceeds its MAC. Since MAC for CIs are defined primarily by their effect on WSIM and changes in electrical conductivity, it is also unlikely that Lubrizol 541 and Nuchem PCI-105 would meet the requirements that 1.5 times the REC not give a WSIM value less than 70 nor result in a change greater than 40 percent in electrical conductivity of a fuel containing static dissipator additive.

One other CI, Tolad 245, only marginally meets the requirements of the proposed revision. The REC was determined to be 21.1 g/m³ (highest, second only to Tolad 249) thus making its MEC 31.6 g/m³. MAC is set at 31.5 g/m³ by QPL 25017-15. Of the three additives failing to meet the proposed lubricity requirement for MIL-I-25017D, only Tolad 249 is among those CI most commonly used by the AF according to a 1984-1985 survey shown in Figure 9.

An important aspect to be considered in incorporating a lubricity requirement into the current military specification is the need for a specific reference fluid. Under the suggested guidelines for a revised MIL-I-25017D specification, passing or failing a particular product may be dependent on the test fluid in which it is evaluated. Fuel properties are not likely to remain constant in future years. Therefore, for the purpose of approving a candidate CI for the QPL, the reference fluid should be a relatively pure hydrocarbon of a known, consistent, composition. Isopar M meets the above requirements. Its composition is well known and there is a considerable data base available for Isopar M in terms of lubricity testing. Currently, Isopar M containing 30 ppm DCI-4A is used as the primary reference fluid and neat Isopar M, containing no additive, is used as the secondary reference fluid in the standard BOCLE test procedure.

TABLE 3.
EFFECT OF REDEFINING RELATIVE EFFECTIVE CONCENTRATION
IN JP-4

Corrosion Inhibitor	g/m ³					
	Redefined REC (0.60 WSD)	QPL REC	Redefined MEC (1.5× REC)	QPL MEC	QPL MAC	WSD at MAC
IPC-4410	5.3	6	8.0	9.0	22.5	0.48
NALCO 5405	6.8	6	10.2	9.0	22.5	0.50
IPC-4445	9.0	6	13.5	9.0	22.5	0.50
P-3305	8.3	9	12.4	13.5	31.5	0.52
NALCO 5403	9.0	6	13.5	9.0	22.5	0.53
UNICOR J	6.8	6	10.2	9.0	22.5	0.53
MOBILAD F-800	6.8	6	17.0	9.0	22.5	0.52
TOLAD 245	21.1	15	31.6	22.5	31.5	0.54
DCI-6A	8.3	6	12.4	9.0	22.5	0.54
LUBRIZOL 541	13.6	6	20.4	9.0	15.0	0.54
DCI-4A	9.0	6	13.5	9.0	22.5	0.55
HITEC E-580	8.3	6	12.4	9.0	22.5	0.55
PRI-19	14.3	6	21.4	9.0	22.5	0.56
TOLAD 249	>22.6	6	>33.9	9.0	22.5	0.61
WELCHEM 91120	8.3	6	13.5	9.0	22.5	0.52
NUCHEM PCI-105	12.8	12	19.2	18.0	18.0	0.54

Notes:

- (1) REC = Relative Effective Concentration
- (2) MEC = Minimum Effective Concentration
- (3) MAC = Maximum Allowable Concentration
- (4) 0.60 WSD = Maximum BOCLE WSD advocated by Air Force
- (5) QPL = QPL-25017-15

R18031/11

3. REFINEMENT OF RPHPLC METHOD FOR DETERMINING CI CONTENT IN JET FUELS

a. Method Development Goals

The objective of this investigation was the development of a relatively simple method for quantifying CI content in jet fuels. The overall goal was the development of a method that could be setup and utilized by Air Force quality assurance laboratories and by refineries. Specific goals set for the method were:

- No sample pretreatment be required.
- Applicability to all approved CI.
- Good precision and accuracy.
- Instrumentation be moderate in cost, readily available and not require special expertise in data interpretation.

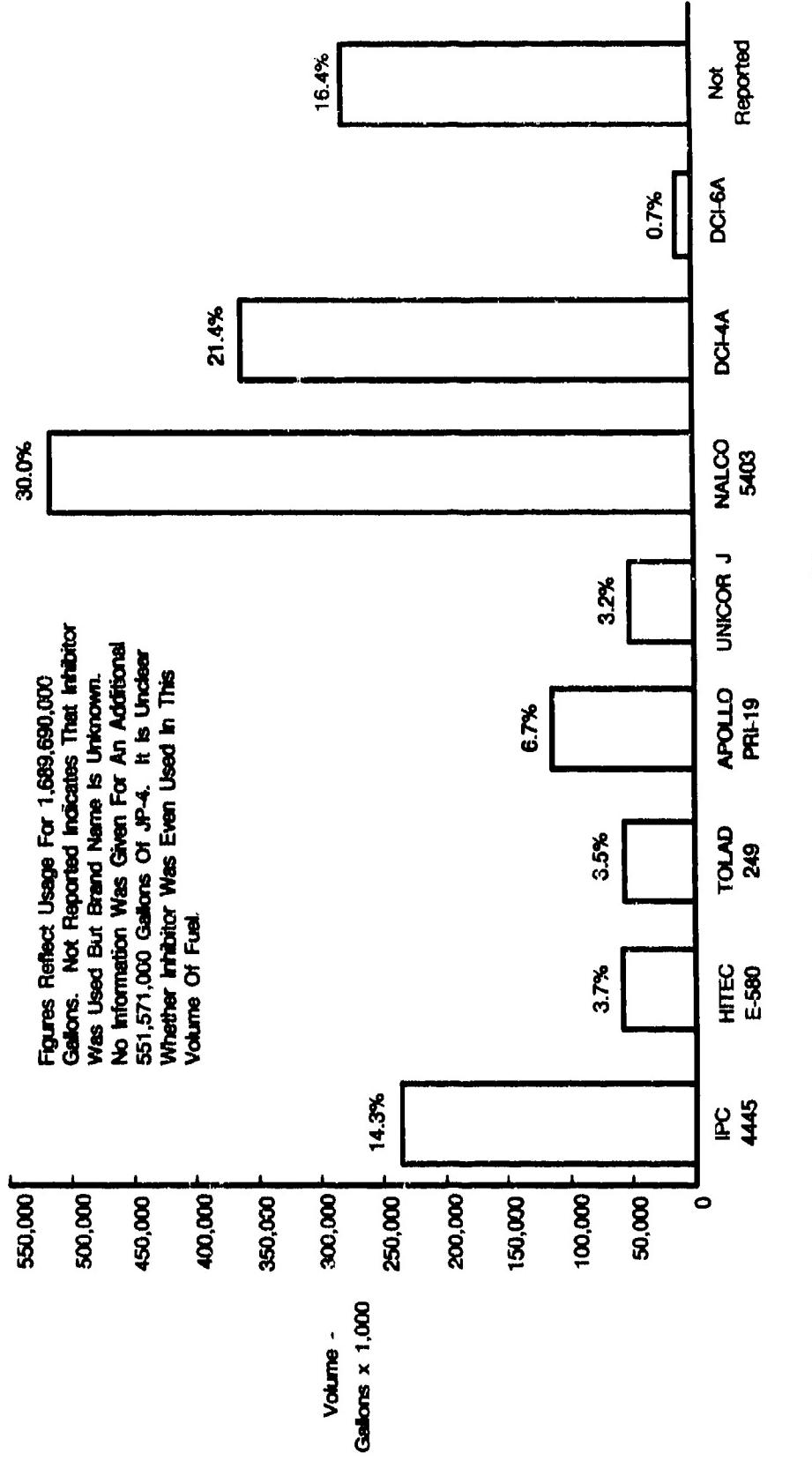


Figure 9. — Air Force Corrosion Inhibitor Usage for JP-4 in FY85

FDA 346167

b. Preliminary Method Development — Theory and Mechanism

The theory upon which the current methodology was developed takes advantage of the unique properties of the CI components and the ability of RPHPLC to resolve those components. All of the approved CI use polar, surface-active organic compounds as active ingredients. Most are similar to the trimer, dimer, and monomer linoleic acids. The material safety data sheets for eleven CI describe active ingredients as high molecular weight organic acids or their derivatives. One exception was Tolad 245, which is described as "acylated glycols and alkanolamines". Four CI claim proprietary ingredients only.

The active ingredients used in CI do not lend themselves well to gas chromatography because of their low volatility and would require derivitization prior to analysis. Other methods such as infrared spectrometry require extraction of the CI from the fuel matrix prior to detection. In addition, infrared spectroscopy requires expert data interpretation.

However, considering the ionic nature and the molecular size of the CI components, it appeared possible to use liquid chromatography for separation from the fuel matrix prior to detection. An ultraviolet detector was chosen because carboxylic acids absorb fairly well in the ultraviolet region of 200 to 210 nanometers. This type of detector is very stable, sensitive, and readily available.

The preliminary method, described in Reference 8, demonstrated that polar high molecular weight (MW) compounds such as the model compound dilinoleic acid, could be separated from a fuel matrix and quantified down to about one ppm. This was accomplished using a cyanopropyl bonded phase column and a mobile phase composed of 60 percent isopropanol and 40 percent buffer (pH 7.0, 0.395 M $\text{KH}_2\text{PO}_4/\text{NaOH}$). This mode of HPLC analysis is termed reverse phase because the mobile phase is more polar in nature than the column stationary phase. Normal phase HPLC is just the opposite.

The apparent separation mechanism of the cyanopropyl column is size exclusion of ionically neutralized CI components. Size exclusion describes the mechanism by which sample molecules elute through a column stationary phase according to their MW, the largest molecules passing through the column first. The order of elution is trimer linoleic acid (MW 845), dimer linoleic acid (MW 565), and mono linoleic acid (MW 282) followed by the fuel matrix components (MW less than 225). The buffer ionically neutralizes the acids at a pH of 7.0 causing them to elute as narrow zones that increases their detectability. This mechanism is termed ionization suppression (IS). IS is believed to control chromatographic retention by suppressing the ionization of the ionic sample with a mobile phase modifier such as a buffer. IS is most useful in the range of pH 3 to 8 and is normally performed using reversed phase columns (i.e., C-18 (octadecyl), C-8 (octyl), etc.). The result is the elution of compounds exhibiting sharper peaks than peaks produced without the buffer.

In support of this theory, the presence of the $\text{KH}_2\text{PO}_4/\text{NaOH}$ buffer was necessary as no peaks were observed when pure water was substituted. Further evidence is given by capacity factor (k') calculations that can be defined as a measure of chromatographic efficiency. Small k' values indicate that the solute is not well retained by the column packing. The k' for dilinoleic acid was 0.0 and that of linoleic acid was 0.14. This indicates that both compounds are essentially unretained by the column and implies a separation mechanism of size exclusion facilitated by IS. The result is an on-column separation of CI components from the fuel matrix. The sample with no pretreatment is simply injected directly into the chromatograph for analysis.

A limited survey of CI was conducted using the RPHPLC method. Figure 10 is a chromatogram of DCI-4A obtained by this method. The survey indicated that most CI were, in fact, multi-component and that greater chromatographic resolution between the components would be necessary to achieve good precision and accuracy.

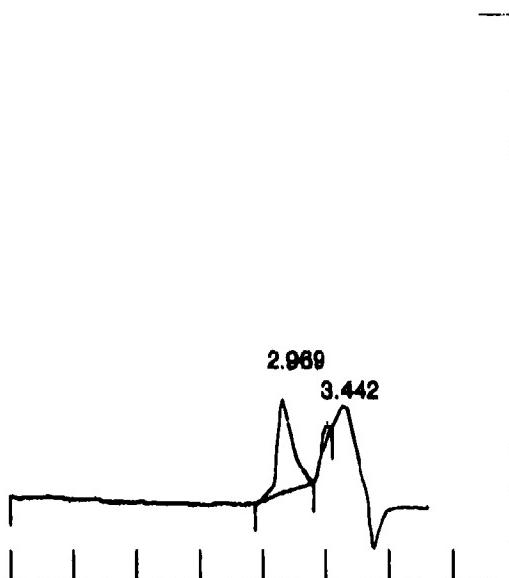


Chart Speed 0.9 cm/min
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FDA 346190

Figure 10. — Chromatogram of DCI-4A Using Preliminary RPHPLC Method

c. Method Modification

This phase of the investigation focused upon modification of the preliminary method to improve the chromatographic resolution of the CI components. Building upon the previous research, it seemed obvious to evaluate the possibility of increasing resolution by increasing the analytical column length. It was found, however, that as column length was increased, the peak widths increased, but with no improvement in resolution. Adjustments in the mobile phase also failed to provide the needed resolution.

Various other column types were coupled after the cyanopropyl column to evaluate their effect on CI peak resolution. Amino, phenyl, C-18 and C-8 bonded phases were rigorously evaluated, including ion pairing techniques, but no increase in CI component resolution was obtained. In fact, it became apparent that adjustments made to increase k' of the components by mobile phase or column-type changes either had no effect, or so dramatically increased k' that the CI components were lost in the fuel matrix peak.

Ion exchange chromatography (IEC) was also evaluated. Typically, IEC is difficult to use because of the many variables that can affect results. It does, however, offer the capability to precisely vary the chromatographic retention of ionic compounds. The first column investigated was a Particil 10 SAX strong anion exchange (Whatman). Use of this column resulted in too much retention of the CI compounds that caused them to be lost in the fuel matrix peak. Attempts to decrease retention by varying the mobile phase parameters proved unsuccessful. Results did indicate, however, that weak ion exchange columns could likely provide the desired resolution. Subsequently, a carboxymethyl weak ion exchange column (Toyo Sota Co.) was coupled to the cyanopropyl column. Mobile phase ionic strength, pH, and percent organic modifier were varied and evaluated for optimum conditions. DCI-4A in clay treated JP-4 was used to evaluate the test parameters. DCI-4A was chosen because of its complex nature.

Information from DuPont Co. indicated that DCI-4A was about 16 percent high polymers or trimer acids, 53 percent dimer acids, 18 percent monomer acids, and 13 percent rosins. It is a by-product of pine tree cellulose processing.

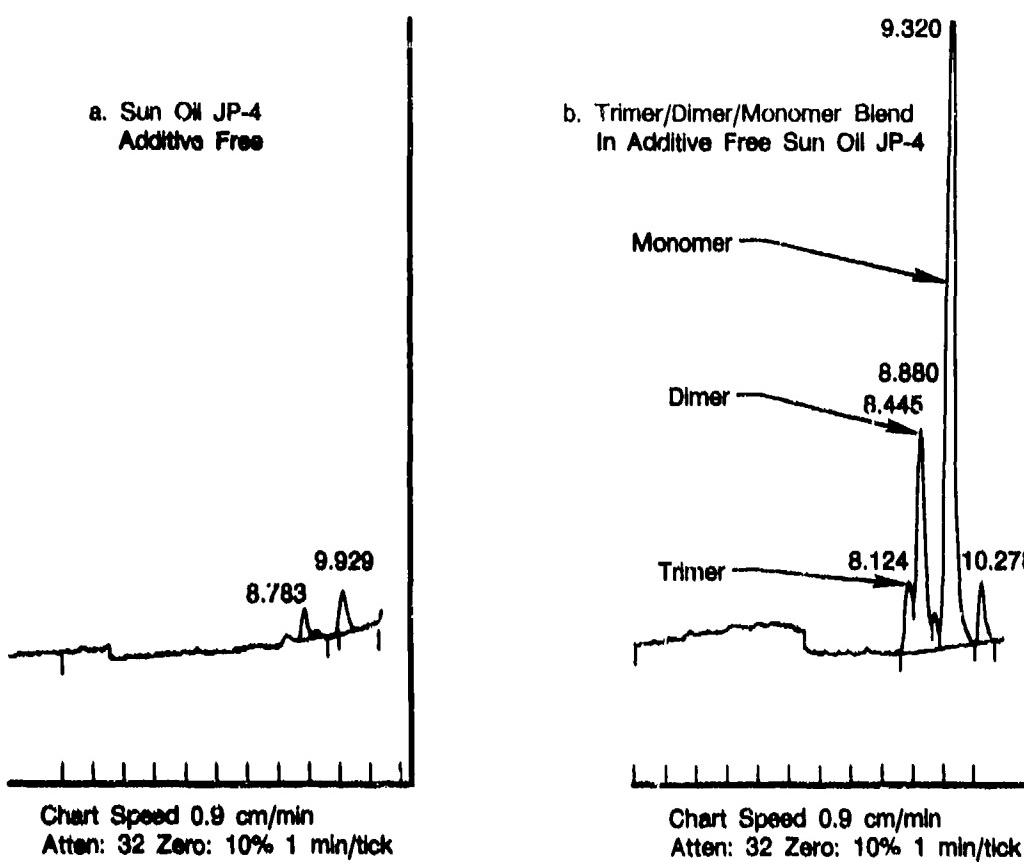
The low percentage of trimer acid in DCI-4A caused its peak to appear only as a leading shoulder on the dimer peak at a 32 ppm level of DCI-4A. However, a blend of 23 ppm trimer (TLA), 25 ppm dimer (DLA), and 21 ppm monomer linoleic acid (MLA) produced a very good separation. Figure 11-a shows the chromatogram of the additive-free JP-4 used to make the blend while Figure 11-b shows the separation achieved for the trimer, dimer, and monomer peaks. Very good resolution ($R=0.9$) of the trimer acid from the dimer acid was obtained. Previously, the trimer and dimer acids coeluted first, followed by coelution of the monomer acids plus the rosins and polar fuel components.

At this point, several observations were made that caused reconsideration of which additive components or peaks should be used for calibration. These observations are summarized below:

- (1) A review of chromatograms for fuels before and after storage suggested that dimer acid levels may diminish rapidly with time. It appeared that monomer acids, however, diminished at a much lesser rate and may more closely approximate the CI content of a fuel upon receipt of the sample.
- (2) BOCLE data indicated that MLA is an excellent fuel lubricity improver. BOCLE results on three neat JP-4 samples containing 10 ppm MLA, DLA, and TLA, respectively, showed the order of lubricity enhancement to be MLA (0.51mm WSD) greater than DLA (0.55 mm WSD) greater than TLA (0.56 mm WSD).
- (3) A high density fuel sample (87-POSF-2605) received from AFWAL/POSF for determination of fuel lubricity and CI content produced a BOCLE WSD of 0.51 mm. Using the BOCLE curves showing WSD vs CI concentration, a 0.51 mm WSD was indicative of approximately 24 ppm DCI-4A. When calibrating on the dimer peak, RPHPLC analysis indicated a CI content of 9.5 ppm. However, when calibrations were performed on the monomer peak, the CI content measured 22 ppm that agreed significantly better with the BOCLE/CI curves.

One point of concern was that some fuels displayed a small additional peak that coeluted close to the same retention time as the monomer acid peak, and this could be contributing to the integrated peak height of the monomer. The above findings indicated that further method development was necessary to separate the monomer acid peak from the fuel components.

Experience with strong cation exchange chromatography and the information gained with weak ion and strong anion columns, indicated that the use of a strong cation exchange bonded phase column, coupled after the cyanopropyl column, could be made to provide the desired separation. With small mobile phase modifications, the monomer acid peak was resolved from the fuel components as shown in Figure 12 at a retention time of 5.657.



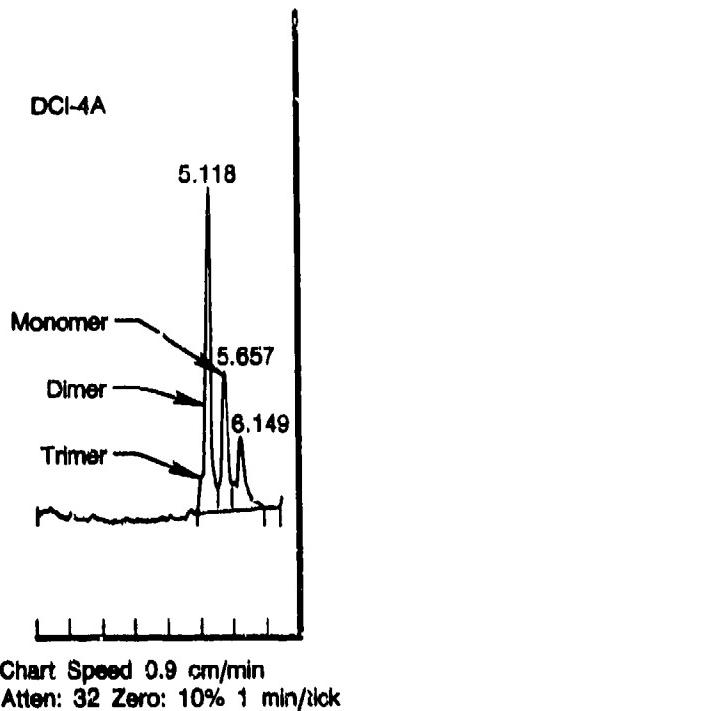
FDA 346191

Figure 11. — Carboxymethyl Column Separation of Dimer, Trimer and Monomer Linoleic Acids in Additive Free Sun Oil JP-4

Although, complete resolution of the monomer peak was achieved, there was some loss in resolution between the trimer and dimer peaks. This loss, however, did not seem to affect quantitation results. Analysis of the 87-POSF-2605 sample was repeated using DCI-4A standards. As shown in Figure 13, calibration on the dimer peak produced a result of 9.2 ppm, while calibration on the monomer peak indicated 24.4 ppm DCI-4A. It was apparent that more than just the fuel peak (probably the rosins) was resolved from the monomer peak since the size of the resolved fuel peak was much greater with DCI-4A additive than without. Apparently, some minor DCI-4A constituents were included in the fuel peak. This peak will now be referred to as "others" to acknowledge this observation. The retention time differences were due to different flow rates (i.e. 0.5 vs 0.75 ml/min.) used during final method optimization. This affected retention times only.

The mechanism of this separation is a form of ionization suppression (IS) as described earlier. The phenylsulfonate bonded phase of the Particil 10 SCX strong cation exchange column acts to provide the reverse phase-like surface normally used in IS; except there is the cationically active sulfonate moiety present. We found that by adjusting the mobile phase pH to 5.5, the ionic strength of the buffer to 0.02 M, and using a methanol organic modifier at 90 volume percent, good separation of the CI components was obtained. We believed that under these conditions, the sulfonate moiety competes to some degree with the trimer, dimer, and monomer acids and the "other" components for the available sodium and potassium cations. This action tends to slightly

increase retention of both the CI and "other" components on the phenyl phase, thus providing the desired separation.



FDA 343192

Figure 12. — Chromatogram of Resolved Monomer Acid Peak

In addition, we believed that only polar molecules and large molecules with molecular weights greater than about 200 are eluted ahead of the fuel matrix peak. As shown in Figure 11, peaks have been observed in additive free fuels with this method. These peaks could be naturally occurring lubricity compounds, auto-oxidation products or other contaminants. Therefore, the method may have applications in the isolation and identification of naturally occurring lubricity agents and in studies of fuel thermal stability.

d. Finalized Test Method

The instrumentation described in Section II was used for all QPL CI evaluations with the exception of the substitution of the 50- μ L sample loop for that of a 20- μ L sample loop on the Rheodyne injector valve. The instrumental conditions used in the final method are given here:

A cyanopropyl bonded phase column (Brownlee Labs), 5 micron particle size, 22 cm \times 4.6 mm with 3 cm cyanopropyl guard column, coupled to a Particil 10 SCX column, 25 cm \times 4.6 mm (Whatman, Clifton, NJ) were used for the separations. It should be noted that the internal diameter of the column connecting tube will affect the CI peak shape as shown in Figure 14. Considerable peak broadening can occur when the tubing is increased from 0.007 inches to 0.01 inches.

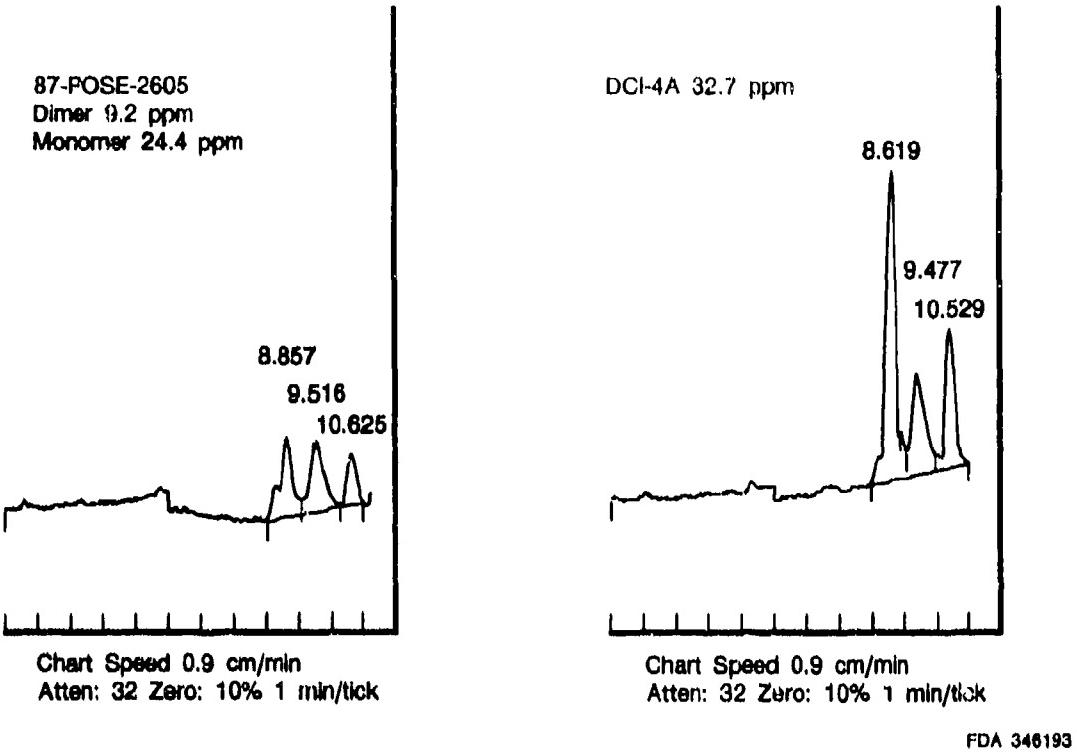
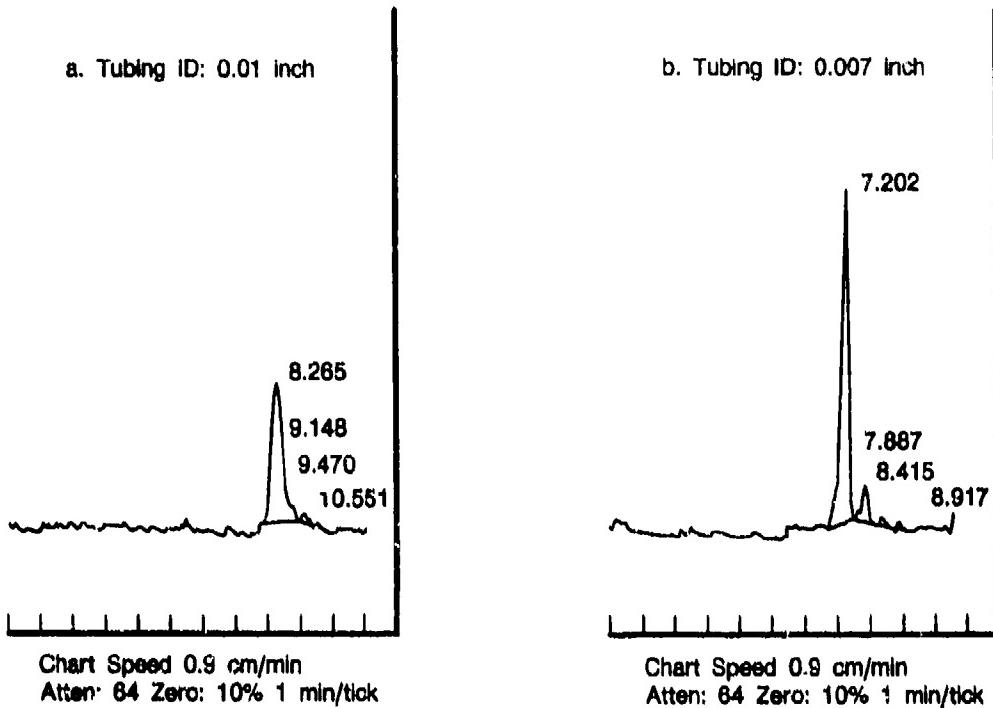


Figure 13. — Effect of Storage on CI Concentration: Stored 87 POSF-2605 Sample Versus New Blend

The mobile phase consisted of 90 percent methanol and 10 percent buffer (pH 5.5, 0.02 M KH_2PO_4 /NaOH). The buffer solution was prepared by diluting 50 mL of 0.1 M KH_2PO_4 and 2.5 mL of 0.1 M NaOH to 250 mL with HPLC grade water. The column heater was set to 30°C (86°F) to eliminate the effect of temperature fluctuations on the chromatographic separation. We found that a 1°C column temperature change altered peak retention times. A temperature of 30°C (86°F) was sufficient to overcome this problem and yet not damage the column packing. In addition, baseline fluctuations and system reequilibration time were reduced with column temperature control.

The standards and samples were syringe injected directly into the HPLC system via the 20- μL sample loop. A 500- μL smooth bore glass and Teflon syringe was best for this purpose. The sample loop was first washed with a 500- μL aliquot of sample from the syringe. The second 500- μL aliquot was the analysis sample. This assured complete sample loop filling and that sample loss and cross contamination was minimized. Between samples, the syringe plunger was removed and the syringe was thoroughly cleaned with isopropyl alcohol and acetone. It was then dried with clean nitrogen.

During a chromatographic analysis, the additives eluted first, ahead of the fuel matrix. After the CI products and the bulk of the fuel had eluted, it was necessary to remove the rest of the fuel sample from the column prior to the next analysis. To accomplish this, a programmed wash cycle using the ternary reservoir capability of the chromatograph was used. Table 4 shows time, reservoir, percent, and flow rate in mL/minute. Reservoirs A, B and C contained isopropanol, methanol/buffer solution, and HPLC grade water, respectively. Analysis time, including wash and reequilibration of the columns, was 35 minutes.



FDA 346194

Figure 14. -- Effect of Connecting Tubing I.D. on Peak Broadening

TABLE 4.
COLUMN WASH GRADIENT PROGRAM

Time	Reservoir			Flow
	%A	%B	%C	
0.00	0	100	0	0.75
8.00	0	100	0	0.75
8.50	0	0	100	1.0
9.00	50	0	50	
9.50	50	0	50	
10.00	100	0	0	
16.00	100	0	0	
16.50	0	0	100	
17.00	0	0	100	
17.50	0	50	50	
18.00	0	100	0	
28.00	0	100	0	1.0
28.50	0	100	0	0.75
35.00	0	100	0	0.75

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Prior to shutting the system down, pure water was pumped through the columns for at least five minutes at 0.75 mL/minute followed by 20 minutes of isopropanol at 0.75 mL/minute. This removed all buffer salts and preserved the system and columns against bacterial growth and corrosion.

e. Applicability to QPL-25017-15

Standards were prepared and analyzed at four different concentrations for each QPL CI. These results were subjected to linear regression analyses. Appendix F contains the calibration chromatograms for the 15 approved CI. With the exception of Lubrizol 541, Tolad 245, and Tolad 249 the principal ingredient seems to be dimer acids. Lubrizol 541 appears to contain mainly monomer acid types. Tolad 249 appears to contain a relatively high percentage of trimer acids at a retention time of 4.89 minutes. Tolad 245 peak shape and retention times do not correspond to trimer, dimer or monomer acids. This is because the active ingredients are not acids, but "acylated glycols and alkanolamines." It is interesting to note that the method is applicable to these compounds as well. Figures F-16 and F-17 of Appendix F show chromatograms of the CT JP-4 base fuel and of trimer linoleic, dimer linoleic, and monomer linoleic acids, respectively. Comparison of these retention times with those obtained for the various CI additives is helpful in their understanding.

Table 5 contains the SEE, correlation coefficient, and y-intercept for each CI. The worst correlation coefficient was that of IPC 4410 with a 0.993. The best was Tolad 245 at 1.00. SEE varied from a high of 2.0 ppm for IPC 4410 to a low of 0.12 ppm for Tolad 245.

TABLE 5.
LINEAR REGRESSION STATISTICS FOR CI CALIBRATION STANDARDS

CI Product Name	Correlation Coefficient	SEE	Y-Axis Intercept
DCI-4A	0.997	1.17	-2.0
DCI-6A	0.995	1.87	-1.4
HITEC 580	0.999	0.81	-1.9
IPC 4410	0.993	2.00	-4.5
IPC 4445	0.999	0.24	-1.3
LUBRIZOL 541	0.998	0.98	-1.9
MOBILAD F800	0.999	0.64	0.1
NALCO 5403	0.994	1.68	-3.4
NALCO 5405	0.994	1.27	-5.4
PCI 105	0.999	0.74	-1.1
PRI 19	0.997	1.08	-1.8
TOLAD 245	1.000	0.12	-5.5
TOLAD 249	0.997	1.47	0.7
UNICOR J	0.998	1.05	-2.5
WELCHFM 91120	0.999	0.87	-2.7

R19981/II

Extreme care and good analytical technique were required when making up CI standards. The stock standard was prepared by weighing the additive concentrate to four decimal places in a clean Teflon bottle then volumetrically diluted with clay treated or additive free fuel. Serial dilutions into appropriate volumetrically pipetted quantities of fuel diluent, in Teflon, were then made. The pipets used were thoroughly cleaned and then rinsed several times with stock standard prior to making the working standards. It was found that failure to follow this procedure would yield either erroneously high results for unknowns if a single point external standard calibration was made, or a low correlation coefficient and SEE if a calibration line was used.

The type fuel used as diluent did not appear to matter. Good results were obtained using CT JP-4 standards to analyze JP-5 and JP-8X type fuels. However, until more experience is gained, the use of clay treated fuels of the type to be analyzed for standards preparation is recommended.

f. Chromatograms

Upon first inspection, the chromatograms in Appendix F appear to have unique fingerprints by which each additive might be identified. Close examination, however, reveals that many have essentially the same fingerprints, especially at lower concentration levels. Further, the spectra will probably be altered after the effects of storage and transport have acted upon the CI/fuel blend. The dramatic effect of CI depletion can be seen in Figure 13, which compares the chromatogram of a DCI-4A fuel blend subjected to short term storage to that of a freshly blended sample.

The use of a single compound such as dilinoleic acid to quantify these additives does not appear to be possible. The UV detector responses to different CI at the same concentration do not correlate well when calibrating on the DLA peak alone. There are a number of possible explanations for this lack of correlation:

- Differences in percent active ingredient between CI.
- Variability due to different isomeric forms of dimer linoleic acid. Emery Chemical Co. indicated that they could not verify the molecular structure of their 97 percent pure dilinoleic acid used in this study. The dimer linkage can, apparently, be very complex.
- Dimer acids, other than linoleic, could be used as active ingredients in CI. These dimer acids, differing in carbon number or placement of the double bonds could have similar chromatographic retention times, but produce different UV detector responses.

It is, therefore, necessary to know what additive was blended into a fuel in order to obtain the best analytical results. This would limit the accuracy of determinations made on co-mingled bulk storage tanks where several different CI may be present. However for quality assurance testing conducted by suppliers (i.e., refiners) and users (e.g., Air Force bases), this is not considered a severe limitation since, in most cases, the type of CI added to a fuel is known by the user.

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

The technical effort performed under this program fulfilled the need to reevaluate CI in terms of lubricity enhancement and refine the RPHPLC method for determining CI content in jet fuels. It also produced functional references, working curves and spectral fingerprints, to assist in predicting and circumventing potential lubricity related fuel system problems. The work resulted in the following accomplishments:

- Establishment of the concentration required for each QPL CI to provide minimum lubricity improvement based on a 0.60 mm WSD.
- Generation of polynomial curve fits profiling CI efficiency in jet fuels.
- Comparison of maximum lubricity improvement attainable for each CI at maximum allowable concentration.
- Approach for incorporating a lubricity requirement into MIL-I-25017.
- Refinement of the RPHPLC method and determination of its applicability to QPL CI.
- Generation of an RPHPLC spectral library of QPL CI.

1. CORROSION INHIBITOR EVALUATIONS

Conclusions drawn from lubricity evaluations of the CI approved under MIL-I-25017D included the following:

- In general, fuel type has little effect on CI performance.
- Temperature appears to have a significant effect on fuel lubricity. Under standard operating conditions, however, assessment of these effects are beyond the current capabilities of the POCLE.
- CI have unique performance profiles in terms of the concentrations required to achieve acceptable lubricity and the maximum lubricity improvement attainable at maximum allowable concentrations.
- Effective CI concentrations for lubricity improvement range from 6.6 to greater than 22.8 g/m³.
- Maximum effective CI concentrations, at which no further reduction in WSD is realized, range from 9 to 31.5 g/m³.
- Only six of the CI evaluated exhibit acceptable lubricity improvement at the 'minimum effective concentration' levels defined by QPL-25017-15.

- CI fall into three distinctive performance groups in relation to total reduction in WSD achieved at maximum allowable concentration and concentration required to achieve a 0.60 mm WSD.
- IPC-4410 was found to be the most effective CI at improving the lubricity properties of jet fuels, while Tolad 249 was found to be the least effective.

Recommendations for further study include the selection of a specific reference fluid for the purpose of approving CI for the QPL. It is also recommended that future work evaluate the currently approved CI in the selected reference fluid at a 1000-g applied load in accordance with the latest CRC revision of the BOCLE test procedure. It is proposed that performance profiles be generated over a range of nine concentrations and the relative effective concentration be established. A correlation between previous work conducted at 500-g load and that performed at the 1000-g load should be presented.

2. PROPOSED MIL-I-25017 LUBRICITY REQUIREMENT

A thorough review of MIL-L-25017, current Air Force quality assurance requirements, and fleet support needs resulted in the following conclusions:

- A lubricity requirement can be easily incorporated into MIL-I-25017 with minimal changes to the current specification and no significant effect on other criteria used in qualifying candidate CI.
- Inclusion of the lubricity requirement can be most readily accomplished by redefining REC while leaving the requirements for MEC and MAC unchanged.
- The effect of redefining REC is that MEC would be increased for all but two of the currently approved CI.
- Three CI (Tolad 249, Lubrizol 541, and Nuchem PCI 105) would be disqualified based on the proposed MIL-I-25107 revision. A fourth CI, Tolad 245, exhibited marginal performance and should be closely scrutinized before requalifying.

3. REFINEMENT OF RPHPLC METHOD

The RPHPLC analytical method has been refined and can quantitatively determine all 15 approved CI in jet fuels. The method requires no sample pretreatment, allowing direct injection of the fuel to be analyzed. The method has been shown to provide good precision and accuracy. Readily available, moderate cost instrumentation is used. Expert data interpretation is not required and the method can be automated if desired. Additionally, the method may be used in quality control for detecting changes in CI concentrations due to losses during transportation and storage. Identification of an unknown CI based on its chromatogram alone, is currently not within scope of this method due to many CI exhibiting essentially the same spectral fingerprint. This is not considered a severe limitation since, in most cases, the specific CI added to a fuel is known by the user.

It is recommended that the RPHPLC test method for CI be evaluated in a second laboratory. This would allow for procedural "loop holes" to be closed, if found. After this, a round-robin evaluation of the method should be conducted prior to its general use.

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APPENDIX A
ISOPAR M PROPERTY DATA SHEET

TYPICAL PROPERTIES

The values shown here are representative of current production. Some are controlled by manufacturing specifications, while others are not. All of them may vary within modest ranges.

Solvency		Test Method	General Properties (cont.)		Test Method
Aniline point, C(F)	89 (192)	ASTM D611	320-329m	<0.08	
Solubility parameter	7.3	Calculated	330-350m	<0.06	
Kauri-butanol value	27	ASTM D1133	Color, Baybolt	+30	ASTM D156
			Color stability, 16 hr at 100C (212F)	+30	
Volatility					
Flash point, PM, C (F)	80 (176)	ASTM D93	Gravity, API	49.2	ASTM D287
Fire point, COC, C (F)	98 (200)	ASTM D82	Specific gravity @ 15.6/15.6C kg/m3	0.784	Calculated
Auto-ignition temperature, C (F)	338 (640)	ASTM D286	lb/gal	784	
Flammability limits in air, vol% at 21C (70F)	0.6-6.5	Calculated	Refractive Index, 20C	6.53	Calculated
Distillation, C (F)		ASTM D86	Viscosity		ASTM D445
IBP	207 (406)		cP at 25C	2.46	
5%	212 (410)		cP at 100C	0.72	
10%	213 (415)		cSt at 0C	6.80	
50%	213 (434)		cSt at 25C	3.35	
90%	241 (466)		Odor, bulk	very slight	Exxon Method
95%	247 (476)		Odor, residual	none	Exxon Method
Dry point	254 (490)		Odor stability	excellent	Exxon Method
FBP	260 (500)		Freezing point, C (F) <-80 (<-70)		
Vapor pressure, kPa at 0°C	4.1	ASTM D2651	Specific heat, liquid, kJ/kg/C (Btu/lb/F)		
Vapor pressure, psia at 100F	0.6		at 0°C (60F)	205 (0.49)	Calculated from enthalpy data
			at 60C (150F)	2.26 (0.64)	
			at 90C (200F)	2.39 (0.67)	
Composition			Heat of vaporization, kJ/kg (Btu/lb)		
Hydrocarbon type, mass %			at 100C (212F)		
Total saturates	99.5	Mass spectrometer	at BP	307 (132)	Est. from Maxwell's "Data Book of Hydrocarbons" and report of API Project 44 (1953)
Aromatics	0.4	UV Analysis		24 (105)	
Trace compounds					
Sulfur					
Dobrot test	Pass	ASTM D484	Surface Properties		
Total sulfur, ppm	1	Microcoulometer	Demulsibility		
Peroxides, ppm	<1	Exxon Method	Interfacial tension, dynes/cm at 25C	51.0	ASTM D971
			Surface tension dynes/cm at 25C	24.8	du Noy
General Properties					
Average molecular weight					
Bromine index (1)	191	Cryogenic	Toxicological Data		
Copper corr., 1/2 hr at BP	230	ASTM D2710	Inhalation, TLV(2) ppm	300(3)	
Unsulfonated residue, vol%	2	ASTM D130	Acute Oral LD50 (Rat), µg/kg	>10	
UV absorbance 260-319 m	99+	ASTM D483	Acute Dermal LD50 (Rabbit), µg/kg	>3.1	
		FDA Method			
		<1.5	21 CFR 172.882		

(1) Bromine index = Bromine number \times 1000

(2) TLV is a registered trademark of the American Conference of Governmental Industrial Hygienists. It is the threshold limit value or occupational exposure limit—the time weighted average concentration for a normal 8-hour workday, 40-hour workweek, to which nearly all workers may be exposed repeatedly without adverse effect. Refer to the most recent Material Safety Data Sheet for the latest recommended maximum exposure limit.

(3) A TLV has not been established for this product. The value shown has been recommended by Exxon Corporation Medical Research based on consideration of available toxicological data. Additional data are being obtained to help define a recommended occupational exposure limit more conclusively.

Qualifications Certified

January 1987

QPL-25017-15

Superceding

QPL-25017-14

11 April 1984

FSC 6850

**APPENDIX B
QUALIFIED PRODUCTS LIST
OF
PRODUCTS QUALIFIED UNDER MILITARY SPECIFICATION
MIL-I-25017**

This list has been prepared for use by or for the Government in the acquisition of products covered by the subject specification and such listing of a product is not intended to and does not connote endorsement of the product by the Department of Defense. All products listed herein have been qualified under the requirements for the product as specified in the latest effective issue of the applicable specification. This list is subject to change without notice; revision or amendment of this list will be issued as necessary. The listing of a product does not release the supplier from compliance with the specification requirements.

The activity responsible for this qualified products list is the Air Force, ASD/ENES, Wright-Patterson AFB OH 45433-6503.

The qualified products are listed in the QPL in two categories.

Category 1 additives are approved for use in fuels conforming to VV-F-800, VV-G-1690, MIL-G-3056, MIL-T-5624, MIL-C-7024, MIL-T-25524, MIL-F-25558, and MIL-T-83133.

Category 2 additives are approved for use in fuels conforming to MIL-T-5624, MIL-C-7024, MIL-F-25558, and MIL-T-83133.

The QPL lists the Government designation, which is also the additive identification, the approving office and date of the letter of approval, the manufacturer's name and address, and additive specifications.

<u>Government Designation</u>	<u>Manufacturer's Designation</u>	<u>Test or Qualification Reference</u>	<u>Manufacturer's Name And Address</u>
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Category 1

PRI-19

Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 9 Apr 85	Apollo Technologies International Corp. 130 Speedwell Ave Morris Plains NJ 07950
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		Plant: c/o
Density at 15°C (kg/)	0.88-0.92		Kramer Chemical Inc. Atlantic Ave and Delaware River Camden NJ 08104
Viscosity (centistokes at 37.8°C)	80-120		
Flashpoint (0°C, minimum)	60		
Neutralization number	100-120		
Ash content (% , maximum)	0.10		
Pour point (°C, maximum)	-18		

<u>Government Designation</u>	<u>Manufacturer's Designation</u>	<u>Test or Qualification Reference</u>	<u>Manufacturer's Name And Address</u>
DCI-4A			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	E.I. duPont deNemours and Company C&P Dept Specialty Chemicals Div Wilmington DE 19898
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.93-0.95		
Viscosity (centistokes at 37.8°C)	48-68		
Flashpoint (°C, minimum)	27		
Neutralization number	100-124		
Ash content (% , maximum)	0.10		
Pour point (°C, maximum)	-18		
DCI-6A			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.93-0.95		
Viscosity (centistokes at 37.8°C)	40-60		
Flashpoint (°C, minimum)	27		
Neutralization number	120-150		
Ash content (% , maximum)	0.10		
Pour point (°C, maximum)	-18		

<u>Government Designation</u>	<u>Manufacturer's Designation</u>	<u>Test or Qualification Reference</u>	<u>Manufacturer's Name And Address</u>
HITEC 580			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	Ethyl Petroleum Additives Division 20 S. Fourth Street St Louis MO 63102-1886
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.91-0.94		
Viscosity (centistokes at 37.8°C)	120-160		
Flashpoint (°C, minimum)	60		
Neutralization number	80-100		
Ash content (% , maximum)	0.10		
Pour point (°C, maximum)	-18		
LUBRIZOL 541¹			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 22 Mar 84	Lubrizol Corporation PO Box 428 Painesville OH 44077
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	15		
Density at 15°C (kg/L)	0.94-0.97		
Viscosity (centistokes at 37.8°C)	34-48		
Flashpoint (°C, minimum)	14		
Neutralization number	152-172		
Ash content (% , maximum)	0.10		
Pour point (°C, maximum)	-18		

¹ This additive is approved for use in MIL-G-5572 fuel, also.

<u>Government Designation</u>	<u>Manufacturer's Designation</u>	<u>Test or Qualification Reference</u>	<u>Manufacturer's Name And Address</u>
NALCO 5403			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	Nalco Chemical Company 77011 Highway 90A Sugar Land TX 77478
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.92-0.94		
Viscosity (centistokes at 37.8°C)	25-50		
Flashpoint (°C, minimum)	38		
Neutralization number	70-100		
Ash content (%, maximum)	0.10		
Pour point (°C, maximum)	-18		
TOLAD 245			
Relative effective conc (g/m)	15	AFWAL/POSF Ltr, 15 Aug 83	Petrolite Corporation 369 Marshall Ave St Louis MO 63118
Minimum effective conc (g/m)	22.5		
Maximum allowable conc (g/m)	31.5		Plant: 369 Marshall Ave St Louis MO 63119
Density at 15°C (kg/L)	0.94-0.96		
Viscosity (centistokes at 37.8°C)	7-14		
Flashpoint (°C, minimum)	32		
Neutralization number	50-62		
Ash content (%, maximum)	0.10		
Pour point (°C, maximum)	-18		

<u>Government Designation</u>	<u>Manufacturer's Designation</u>	<u>Test or Qualification Reference</u>	<u>Manufacturer's Name And Address</u>
<i>UNICOR J</i>			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	UOP, Inc. Box 5017 20 Algonquin Road Des Plaines IL 60017-5017
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		and Universal-Matthey
Density at 15°C (kg/L)	0.93-0.94		Products (France) S.A.
Viscosity (centistokes at 37.8°C)	65-85		Rue D'epinal Calais 62100 France
Flashpoint (°C, minimum)	52		
Neutralization number	110-126		
Ash content (% maximum)	0.10		
Pour point (°C, maximum)	-18		
<i>Category 2</i>			
<i>IPC 4410</i>			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	ChemLink, Incorporated 16950 Wallisville Rd Houston TX 77049
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.94-0.96		
Viscosity (centistokes at 37.8°C)	220-270		
Flashpoint (°C, minimum)	60		
Neutralization number	130-155		
Ash content (% maximum)	0.10		
Pour point (°C, maximum)	-18		

<u>Government Designation</u>	<u>Manufacturer's Designation</u>	<u>Test or Qualification Reference</u>	<u>Manufacturer's Name And Address</u>
IPC 4445			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 1 Dec 82	
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.91-0.93		
Viscosity (centistokes at 37.8°C)	10-40		
Flashpoint (°C, minimum)	60		
Neutralization number	80-100		
Ash content (%) , maximum)	0.10		
Pour point (°C, maximum)	-18		
MOBILAD F800			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	Mobil Chemical Company Chemical Products Div PO Box 250 Edison NJ 08818
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.84-0.88		
Viscosity (centistokes at 37.8°C)	23-35		
Flashpoint (°C, minimum)	38		
Neutralization number	80-100		
Ash content (%) , maximum)	0.10		
Pour point (°C, maximum)	-43		

<u>Government Designation</u>	<u>Manufacturer's Designation</u>	<u>Test or Qualification Reference</u>	<u>Manufacturer's Name And Address</u>
NALCO 5405			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	Nalco Chemical Company 7701 Highway 90A Sugar Land TX 77478
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.91-0.95		
Viscosity (centistokes at 37.8°C)	40-70		
Flashpoint (°C, minimum)	60		
Neutralization number	115-145		
Ash content (%, maximum)	0.10		
Pour point (°C, maximum)	-29		
NUCHEM PCI-105			
Relative effective conc (g/m)	12	AFWAL/POSF Ltr, 12 Sep 86	NuChem Corp Maple Lane PO Box U Blairstown NJ 07825
Minimum effective conc (g/m)	18		
Maximum allowable conc (g/m)	18		
Density at 15°C (kg/L)	0.89-0.93		
Viscosity (centistokes at 37.8°C)	100-150		
Flashpoint (°C, minimum)	60		
Neutralization number	95-120		
Ash content (%, maximum)	0.10		
Pour point (°C, maximum)	-18		

<u>Government Designation</u>	<u>Manufacturer's Designation</u>	<u>Test or Qualification Reference</u>	<u>Manufacturer's Name And Address</u>
TOLAD 249			
Relative effective conc (g/m)	6	AFWAL/POSF Ltr, 15 Aug 83	Petrolite Corporation Industrial chemicals Group 369 Marshall Ave St Louis MO 63119
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.89-0.93		
Viscosity (centistokes at 37.8°C)	7-25		
Flashpoint (°C, minimum)	32		
Neutralization number	85-120		
Ash content (% maximum)	0.10		
Pour point (°C, maximum)	-29		
WELCHEM 91120			
Relative effective conc (g/m)	6		Welchem, Inc. 11200 Bay Area Blvd Houston TX 77507
Minimum effective conc (g/m)	9		
Maximum allowable conc (g/m)	22.5		
Density at 15°C (kg/L)	0.93-0.96		
Viscosity (ccntistokes at 37.8°C)	50-70		
Flashpoint (°C, minimum)	65		
Neutralization number	90-110		
Ash content (% maximum)	0.10		
Pour point (°C, maximum)	0°C		

**APPENDIX C
LUBRICITY TEST DATA
LIST OF TABLES**

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C-1	Effect of Corrosion Inhibitors in ISOPAR M	C-1
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C-4	Effect of Corrosion Inhibitors in Clay Treated JP-5	C-4

Table C-1. Effect of Corrosion Inhibitors in ISOPAR M

Corrosion Inhibitor	Temperature °C	Base Fuel	1.5 ppm	3.0 ppm	6.0 ppm	9.0 ppm	12.0 ppm	15.0 ppm	20.0 ppm	30.0 ppm	42.0 ppm
PRI-19	25	0.80	0.76	0.74	0.71	0.66	0.62	0.61	0.54	0.54	0.54
	75	1.12	1.05	1.03	0.96	0.91	0.88	0.86	0.82	0.76	0.76
HTEC E-580	25	0.75	0.78	0.76	0.76	0.69	0.62	0.60	0.54	0.52	0.52
	75	1.13	0.57	1.05	0.84	0.82	0.85	0.86	0.78	0.73	0.73
DCI-4A	25	0.76	0.74	0.72	0.67	0.60	0.54	0.54	0.50	0.48	0.48
	75	1.01	1.04	0.98	0.90	0.86	0.81	0.68	0.64	0.58	0.58
DCI-6A	25	0.78	0.76	0.76	0.71	0.65	0.60	0.54	0.50	0.48	0.48
	75	1.19	1.08	1.00	0.84	0.90	0.89	0.82	0.76	0.68	0.68
LUBRIZOL 541	25	0.81	0.80	0.78	0.74	0.70	0.68	0.62	0.56	0.54	0.54
	75	1.03	0.98	0.94	0.74	0.76	0.74	0.80	0.77	0.59	0.59
NALCO 5403	25	0.80	0.80	0.72	0.74	0.74	0.61	0.56	0.52	0.52	0.52
	75	1.14	1.08	1.06	0.80	0.81	0.68	0.84	0.73	0.66	0.66
TOLAD 245	25	0.76	0.75	0.76	0.72	0.74	0.70	0.64	0.60	0.55	0.55
	75	1.08	1.13	0.90	0.81	0.83	0.90	0.86	0.66	0.66	0.68
UNICOR J	25	0.80	0.72	0.68	0.64	0.58	0.56	0.52	0.50	0.47	0.47
	75	1.12	0.86	1.02	0.82	0.79	0.82	0.91	0.84	0.77	0.77
IPC 4410	25	0.78	0.77	0.74	0.67	0.62	0.57	0.54	0.54	0.52	0.52
	75	1.08	1.05	0.89	0.80	0.80	0.63	0.88	0.82	0.78	0.78
MOBILAD F800	25	0.74	0.74	0.74	0.70	0.62	0.57	0.55	0.52	0.49	0.49
	75	1.06	0.97	0.87	0.75	0.86	0.85	0.82	0.77	0.70	0.70
NALCO 5405	25	0.76	0.78	0.76	0.72	0.67	0.60	0.56	0.52	0.48	0.48
	75	1.06	0.99	0.97	0.82	0.80	0.84	0.83	0.82	0.69	0.69
TOLAD 249	25	0.78	0.78	0.75	0.73	0.76	0.74	0.72	0.66	0.59	0.59
	75	1.06	1.02	1.02	1.01	0.76	0.72	0.78	0.84	0.82	0.82
P-33C5	25	0.74	0.76	0.76	0.68	0.67	0.56	0.54	0.52	0.47	0.47
	75	1.09	1.06	0.86	0.90	0.89	0.82	0.78	0.77	0.66	0.66
IPC-445	25	0.81	0.78	0.76	0.75	0.72	0.68	0.63	0.58	0.54	0.54
	75	0.94	1.08	1.09	1.07	0.92	1.02	0.92	0.88	0.79	0.79
WELCHEM 91120	25	0.80	0.80	0.78	0.76	0.68	0.62	0.66	0.59	0.56	0.56
NUCHEM PCU-05	25	0.79	0.77	0.77	0.72	0.68	0.66	0.64	0.58	0.55	0.55

en16C

Table C-2. Effect of Corrosion Inhibitors in Clay Treated JP-4

Corrosion Inhibitor	Temperature °C	Base Fuel	1.5	3.0	6.0	9.0	12.0	15.0	20.0	30.0	42.0
			ppm								
ERI-19	25	0.84	0.79	0.74	0.71	0.66	0.64	0.62	0.62	0.56	
	75	0.70	0.57	0.74	0.78	0.66	0.58	0.70	0.62		
MITEC E-580	25	0.83	0.78	0.74	0.67	0.62	0.58	0.58	0.57	0.55	
	75	0.52	—	—	—	0.61	0.68	0.66	0.60	0.56	
DCL-4A	25	0.82	0.78	0.74	0.70	0.66	0.58	0.56	0.54	0.55	
	75	0.78	0.52	0.58	0.63	0.60	0.58	0.58	0.56	0.52	
DCL-6A	25	0.81	0.76	0.76	0.68	0.62	0.59	0.54	0.56	0.54	
	75	0.79	—	—	—	0.72	0.66	0.62	0.58	0.58	
LUBRIZOL 541	25	0.84	0.80	0.77	0.74	0.70	0.64	0.62	0.60	0.54	
	75	0.65	0.62	—	—	0.72	0.57	0.62	0.70	0.66	
NALCO 5463	25	0.78	0.78	0.74	0.65	0.64	0.61	0.60	0.54	0.53	
	75	0.84	—	—	—	0.73	0.69	0.64	0.60	0.56	
TOLAD 245	25	0.81	0.76	0.76	0.72	0.70	0.70	0.68	0.64	0.60	0.54
	75	0.70	—	—	—	0.65	0.52	0.52	0.47	0.60	0.50
UNICOR J	25	0.79	0.75	0.70	0.62	0.61	0.57	0.58	0.52	0.53	
	75	0.86	—	—	—	0.70	0.66	0.63	0.60	0.54	
IPC 4410	25	0.78	0.77	0.69	0.61	0.54	0.53	0.55	0.52	0.48	
	75	0.68	—	—	—	0.65	0.60	0.59	0.56	0.53	
MOBILAD F800	25	0.81	0.76	0.71	0.65	0.62	0.58	0.54	0.54	0.52	
	75	0.67	—	—	—	0.61	0.62	0.62	0.58	0.54	
NALCO 5405	25	0.78	0.78	0.74	0.66	0.60	0.53	0.56	0.51	0.50	
	75	0.70	—	—	—	0.60	0.57	0.55	0.56	0.55	
TOLAD 249	25	0.80	0.78	0.79	0.75	0.70	0.68	0.67	0.63	0.61	
	75	0.83	—	—	—	0.64	0.59	0.58	0.62	0.56	
P-3305	25	0.81	0.80	0.78	0.70	0.61	0.56	0.56	0.55	0.52	
	75	0.60	—	—	—	0.70	0.61	0.58	0.54	0.52	
IPC 4445	25	0.78	0.72	0.86	0.70	0.68	0.53	0.55	0.53	0.50	
	75	0.52	—	—	—	0.56	0.60	0.56	0.53	0.58	
WELCHEM S-120	25	0.81	0.78	0.74	0.68	0.63	0.59	0.56	0.52	0.52	
NUCHEM PC-105	25	0.80	0.76	0.76	0.69	0.66	0.64	0.62	0.58	0.54	

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Table C-3. Effect of Corrosion Inhibitors in Clay Treated JP-8

Corrosion Inhibitor	Temperature °C	Base Fuel	2.5 ppm	3.0 ppm	6.0 ppm	9.0 ppm	12.0 ppm	15.0 ppm	20.0 ppm	30.0 ppm	42.0 ppm
PR-19	25	0.83	0.86	0.80	0.7*	0.70	0.66	0.61	0.59	0.57	0.54
HTEC E-580	25	0.81	0.78	0.75	0.65	0.66	0.61	0.58	0.57	0.55	0.50
DCI-4A	25	0.82	0.76	0.72	0.64	0.61	0.56	0.54	0.52	0.50	0.52
DCI-6A	25	0.80	0.81	0.78	0.68	0.62	0.58	0.56	0.55	0.54	0.56
LUBREZOL 541	25	0.87	0.84	0.80	0.75	0.68	0.64	0.60	0.56	0.56	0.56
NALCO 5403	25	0.82	0.78	0.75	0.65	0.62	0.61	0.56	0.54	0.50	0.50
TOLAD 245	25	0.85	0.79	0.78	0.74	0.70	0.67	0.66	0.62	0.56	0.56
UNICOR J	25	0.82	0.79	0.76	0.68	0.62	0.59	0.54	0.54	0.54	0.54
IPC 4410	25	0.83	0.78	0.70	0.66	0.56	0.56	0.52	0.50	0.48	0.48
MORILAD F800	25	0.87	0.80	0.74	0.58	0.64	0.61	0.57	0.53	0.48	0.48
NALCO 5405	25	0.82	0.77	0.74	0.66	0.62	0.58	0.54	0.53	0.48	0.48
TOLAD 249	25	0.84	0.82	0.82	0.76	0.75	0.70	0.69	0.66	0.60	0.60
P-3366	25	0.77	0.76	0.75	0.70	0.63	0.58	0.57	0.54	0.52	0.520
IPC 4445	25	0.78	0.76	0.75	0.68	0.67	0.62	0.50	0.57	0.54	0.54
WELCHEM 91120	25	0.85	0.80	0.78	0.72	0.66	0.62	0.60	0.58	0.54	0.54
NUCHEM PCI-105	25	0.90	0.86	0.77	0.72	0.65	0.63	0.60	0.57	0.52	0.52

Table C-4. Effect of Corrosion Inhibitors in Clay Treated JP-5

Corrosion Inhibitor	Temperature °C	Base Fuel	ppm						ppm						ppm					
			1.5	3.0	6.0	9.0	12.0	15.0	20.0	30.0	42.0	1.5	3.0	6.0	9.0	12.0	15.0	20.0	30.0	42.0
PRI-19	25	0.80	0.74	0.72	0.74	0.65	0.63	0.60	0.56	0.56	0.52	—	—	—	—	—	—	—	—	—
HITEC E-586	75	0.50	0.50	0.48	0.49	0.52	0.50	0.56	0.60	0.60	0.58	—	—	—	—	—	—	—	—	—
DCL-4A	25	0.76	0.71	0.70	0.64	0.64	0.62	0.59	0.55	0.55	0.51	—	—	—	—	—	—	—	—	—
DCL-6A	25	0.76	0.72	0.58	0.64	0.60	0.52	0.51	0.56	0.52	0.55	—	—	—	—	—	—	—	—	—
LUBREOL 541	75	0.54	0.53	0.51	0.52	0.52	0.50	0.52	0.51	0.50	0.48	—	—	—	—	—	—	—	—	—
NALCO 5403	25	0.49	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
TCLAD 245	25	0.76	0.73	0.73	0.68	0.68	0.63	0.62	0.58	0.57	0.56	—	—	—	—	—	—	—	—	—
UNICOR J	75	0.50	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
IPC 4410	25	0.77	0.74	0.70	0.61	0.60	0.54	0.52	0.49	0.45	0.45	—	—	—	—	—	—	—	—	—
MOBILAD F800	25	0.81	0.76	0.70	0.66	0.57	0.54	0.52	0.52	0.52	0.50	—	—	—	—	—	—	—	—	—
NALCO 5405	75	0.51	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
TCLAD 249	75	0.79	0.76	0.75	0.66	0.61	0.56	0.54	0.54	0.52	0.52	—	—	—	—	—	—	—	—	—
P-3305	25	0.78	0.74	0.76	0.66	0.60	0.56	0.56	0.56	0.56	0.52	—	—	—	—	—	—	—	—	—
IPC-4445	25	0.78	0.78	0.74	0.70	0.64	0.61	0.59	0.58	0.58	0.56	—	—	—	—	—	—	—	—	—
WELCHEM 91120	75	0.74	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
NUCHEM PCI-105	25	0.80	0.75	0.74	0.72	0.68	0.63	0.60	0.59	0.59	0.54	—	—	—	—	—	—	—	—	—

APPENDIX D PERFORMANCE PLOTS

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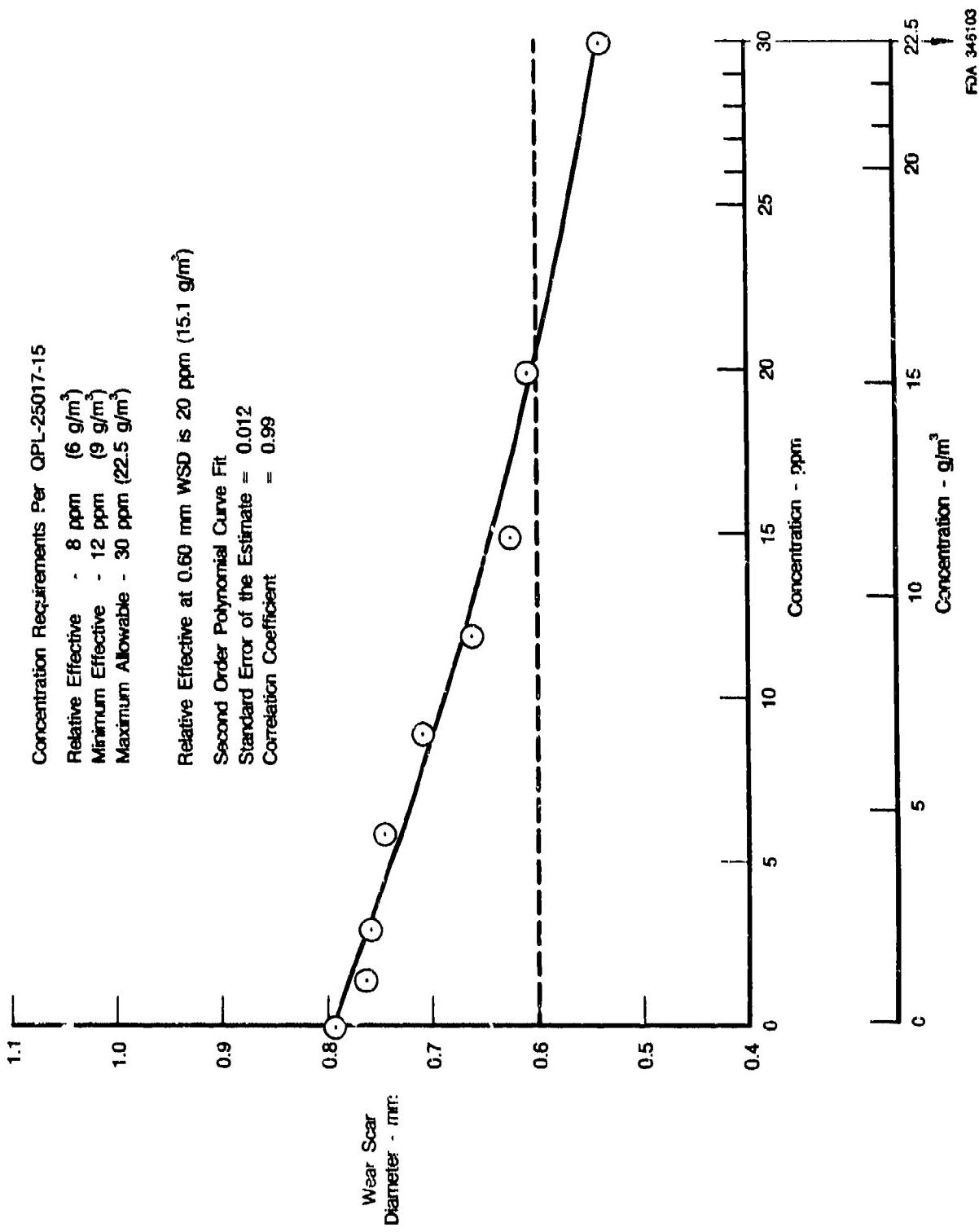


Figure D-1. Effect of Apollo PRI-19 in ISOPAR-M

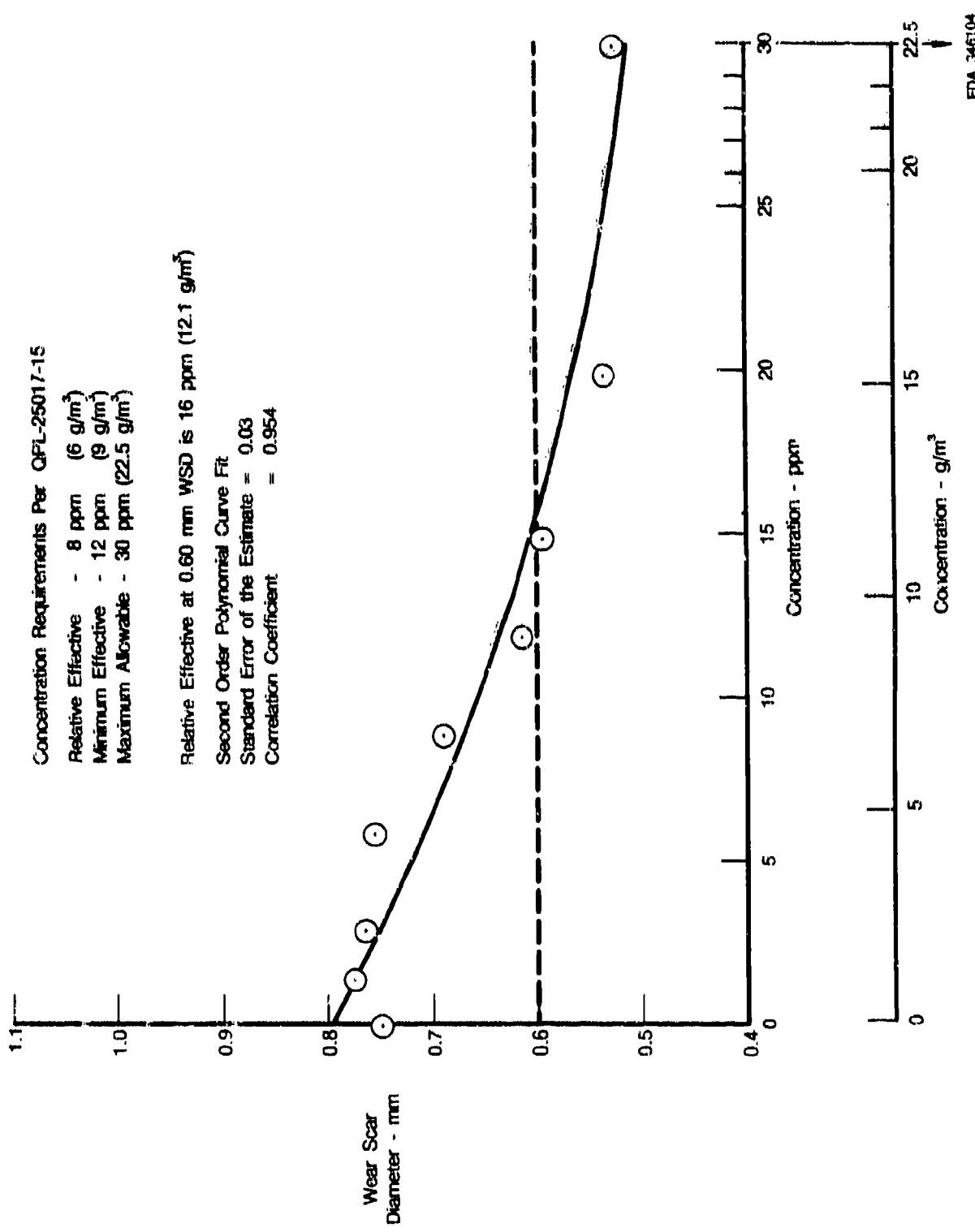


Figure D-2. — Effect of HITEC E-580 in ISOPAR-M

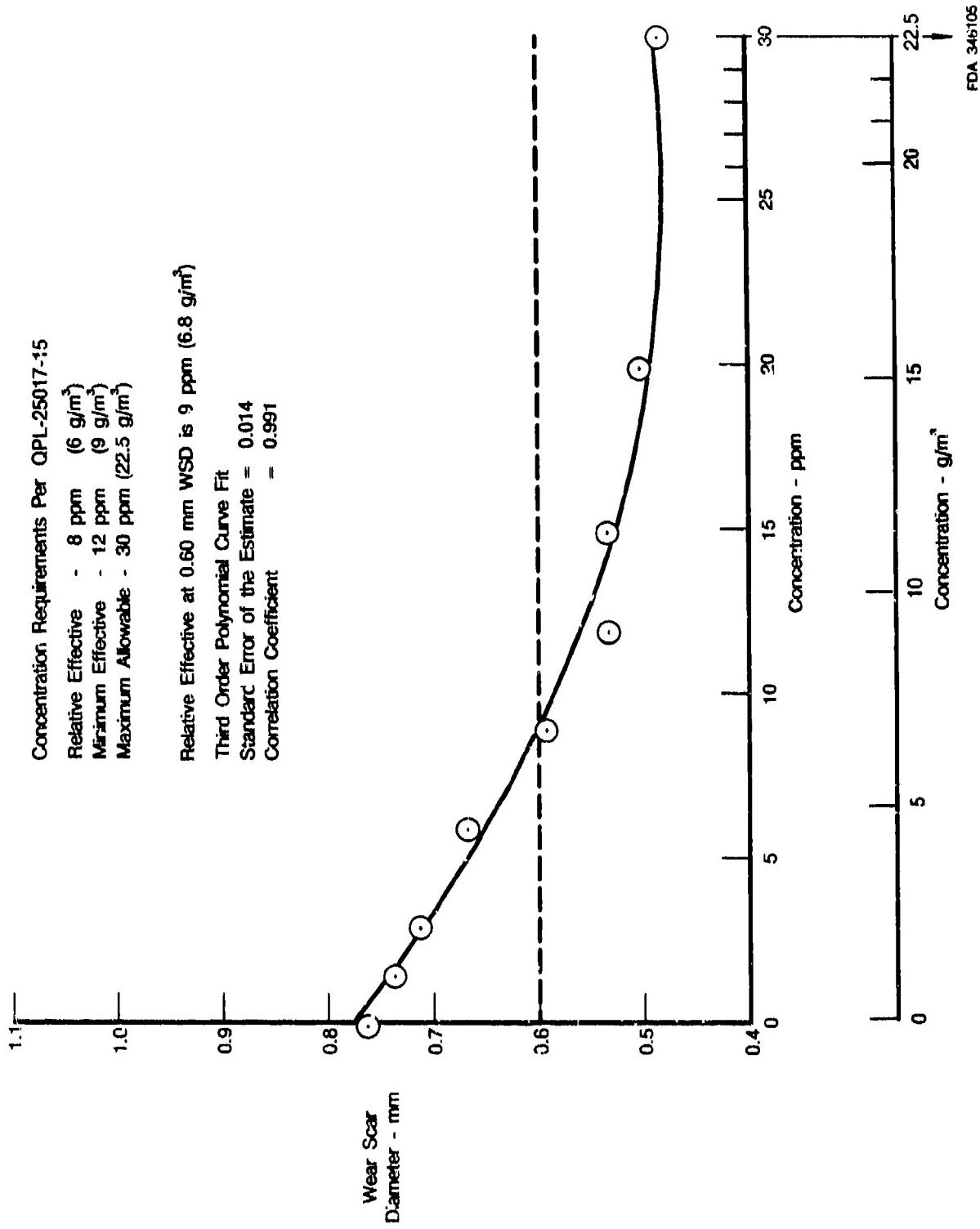


Figure D-3. — Effect of DCI-4A in ISOPAR-M

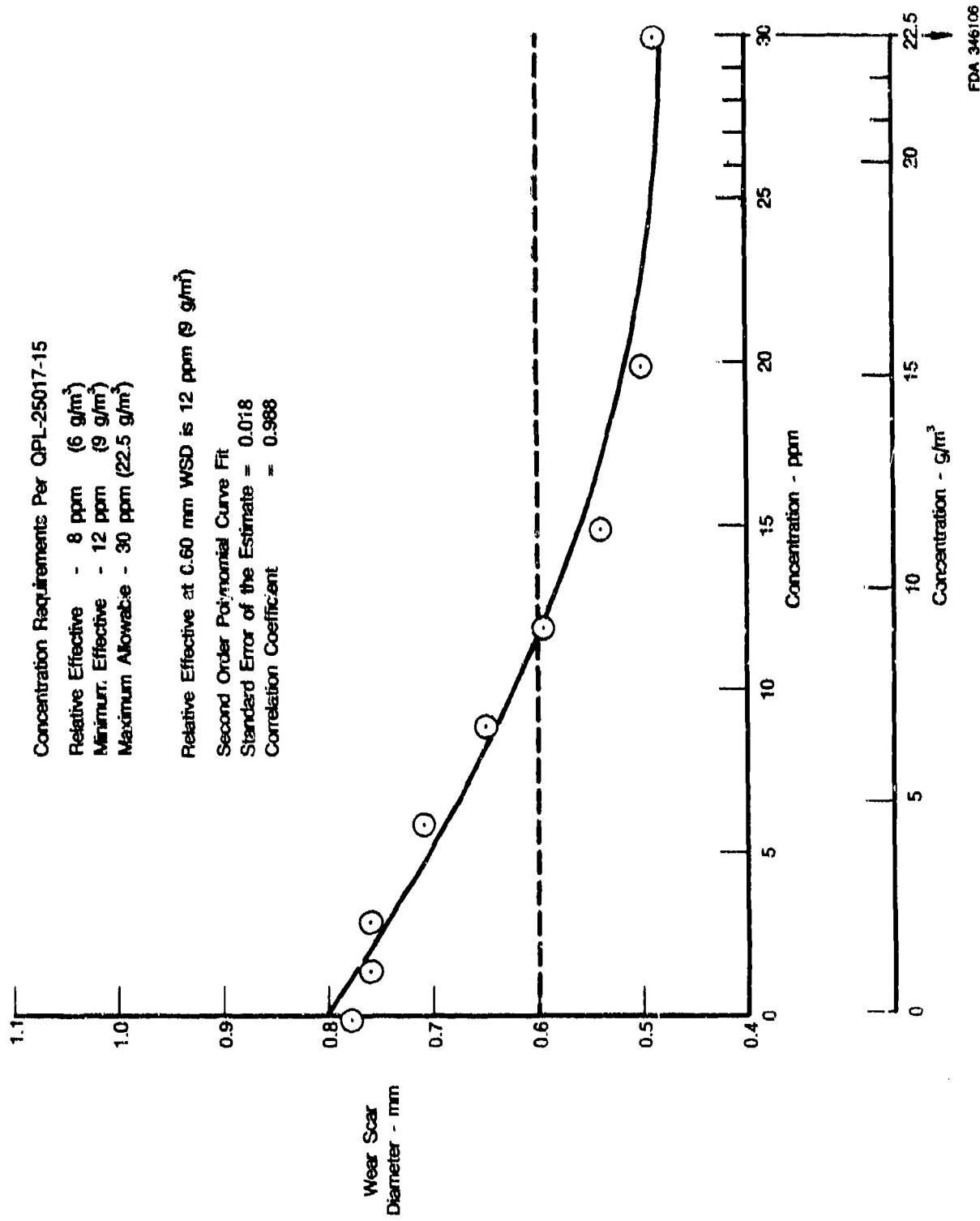


Figure D-4. — Effect of DCI-6A in ISOPAR-M

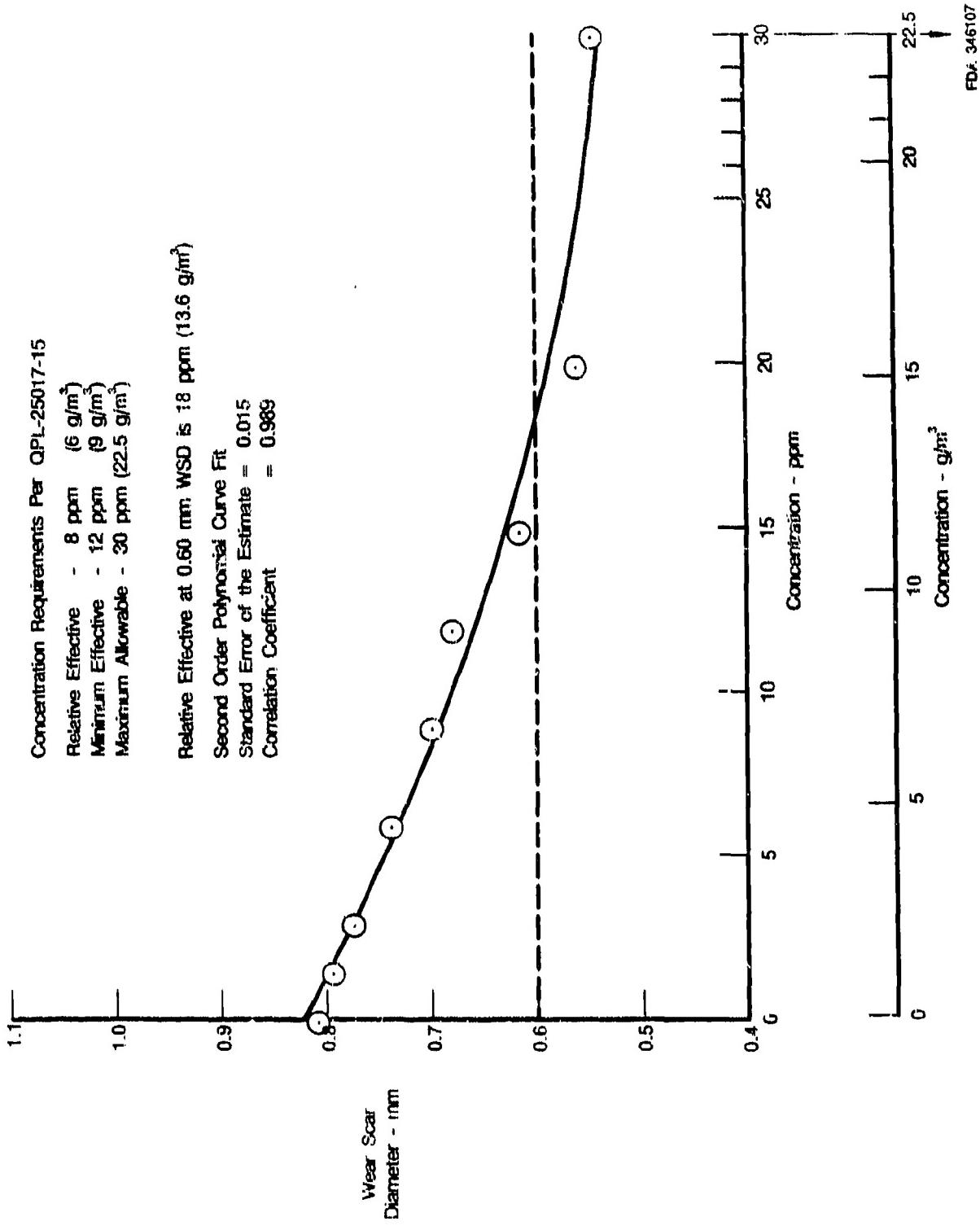


Figure D-5. — Effect of LUBRIZOL 541 in ISOPAR-M

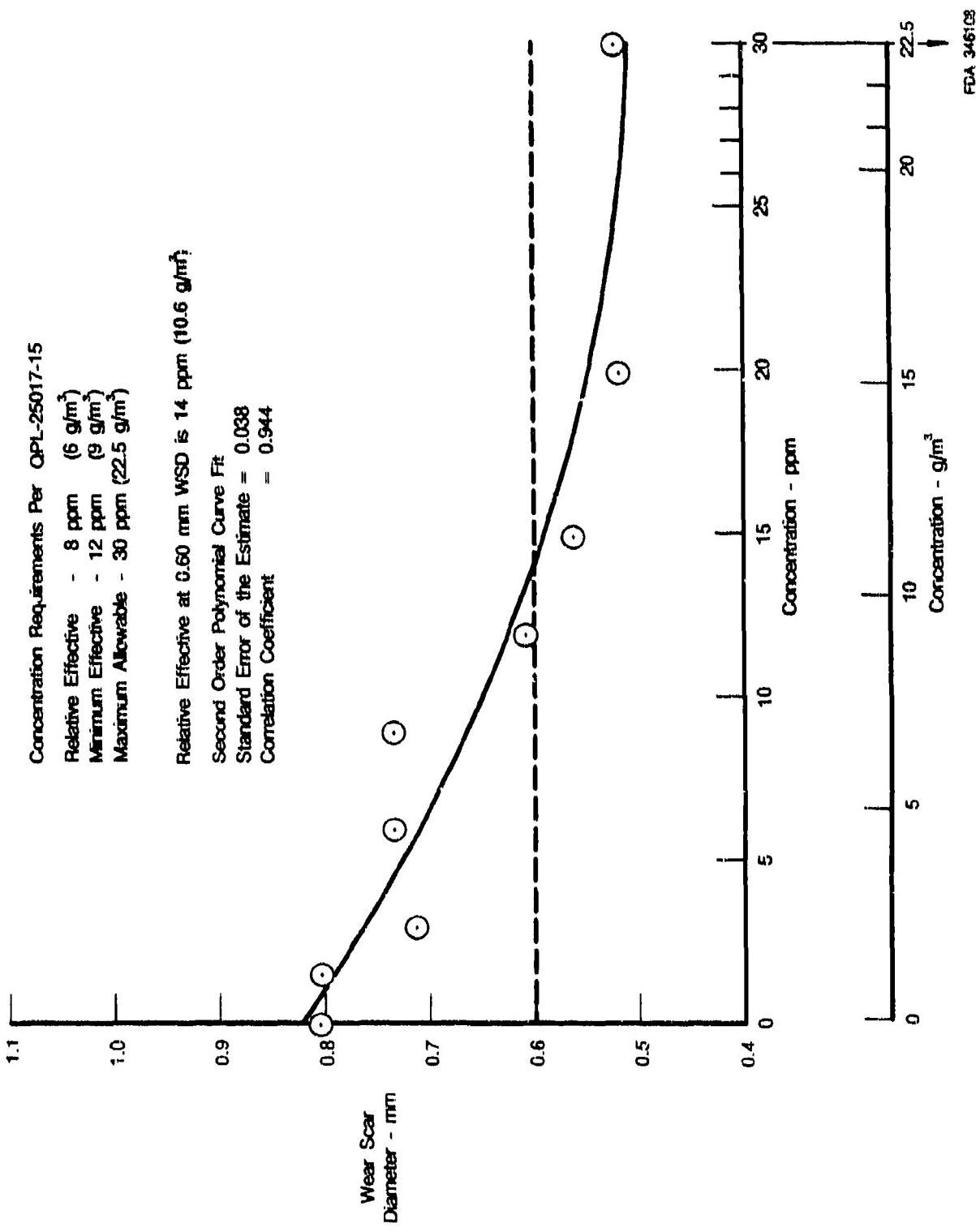


Figure D-6 — Effect of NALCO 5403 in ISOPAR-M

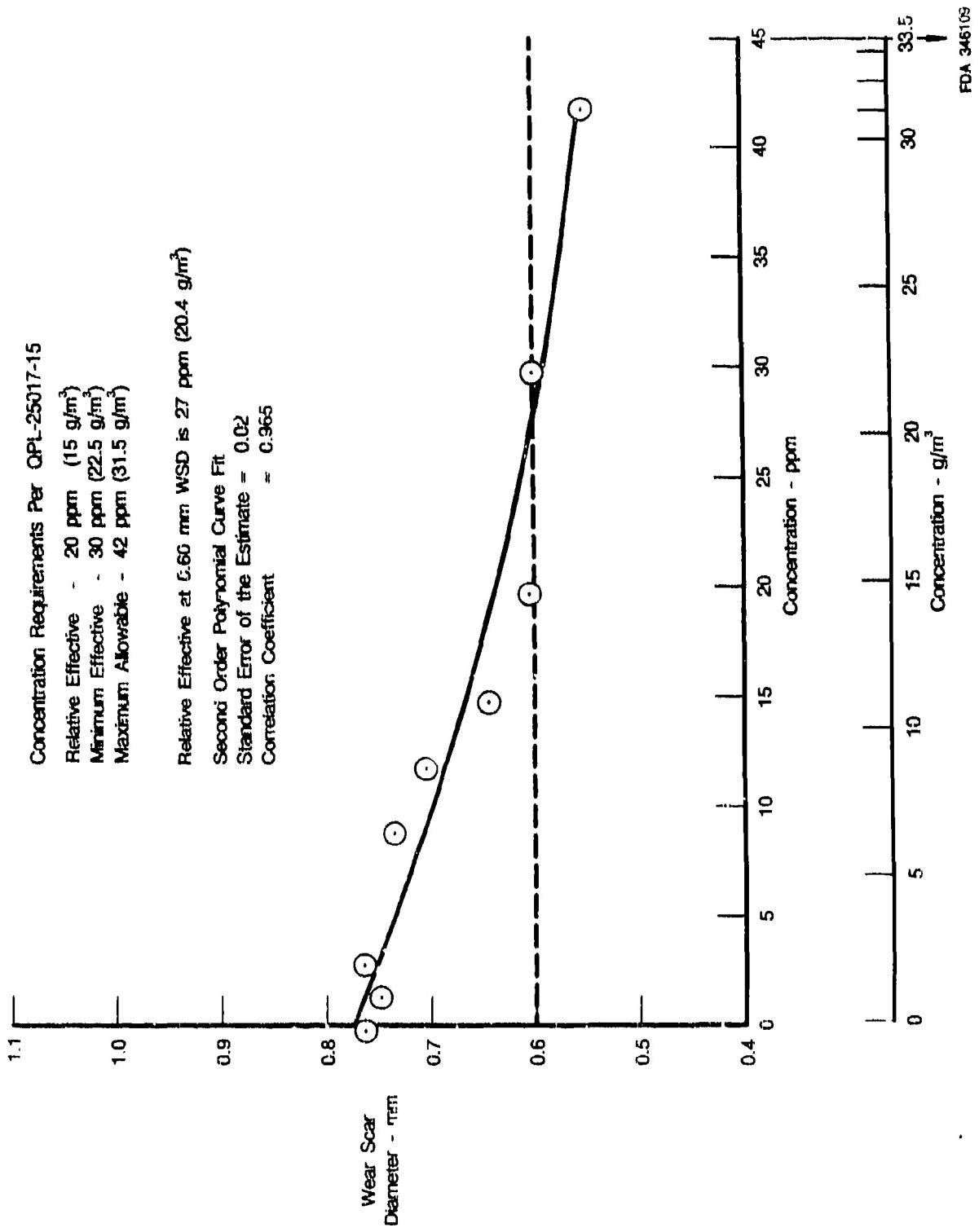


Figure D-7. — Effect of TOLAD 245 in ISOPAR-M

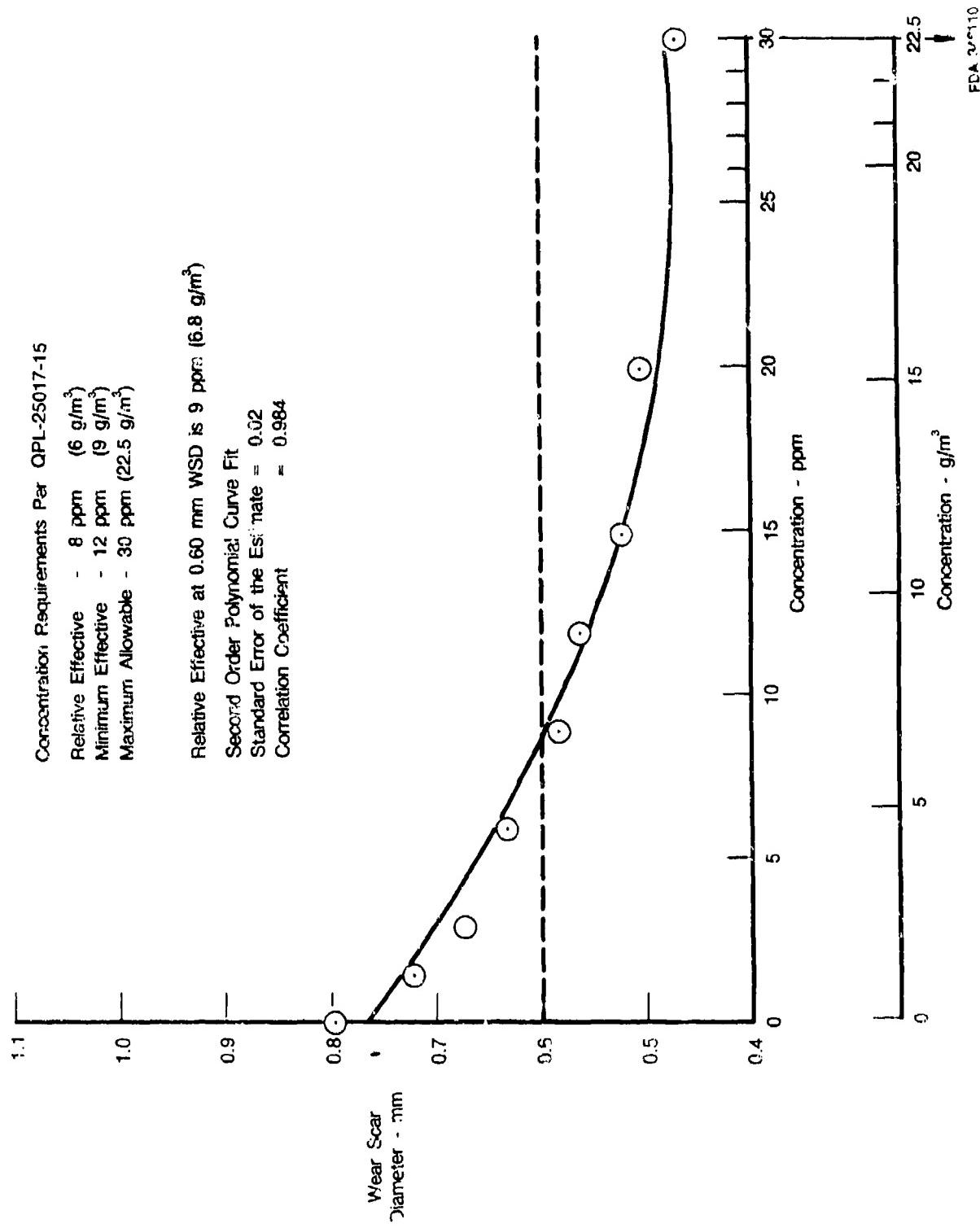
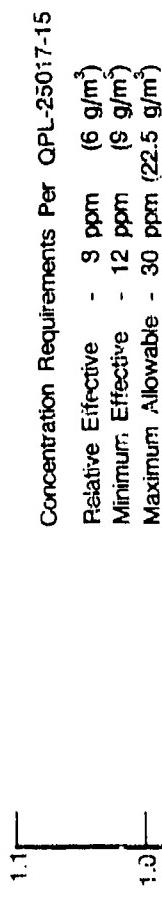


Figure D-8. — Effect of UNICOR-J in ISOPAR.TM



Relative Effective at C.60 mm WSD is 11 ppm (8.3 g/m³)

Third Order Polynomial Curve Fit

Standard Error of the Estimate = 0.014
Correlation Coefficient = 0.992

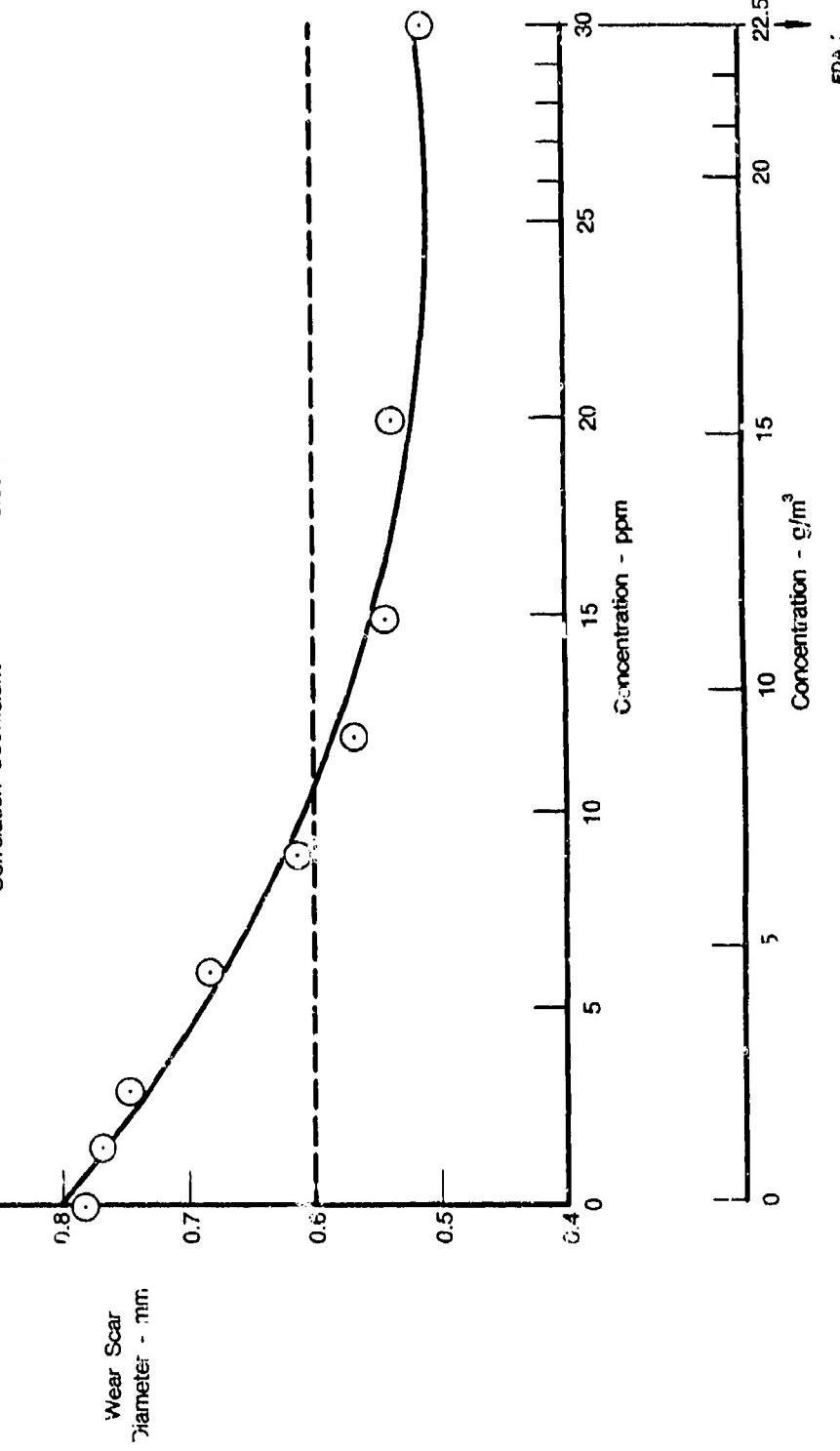


Figure D-9. — Effect of IPC-4410 in ISOPIAR-M

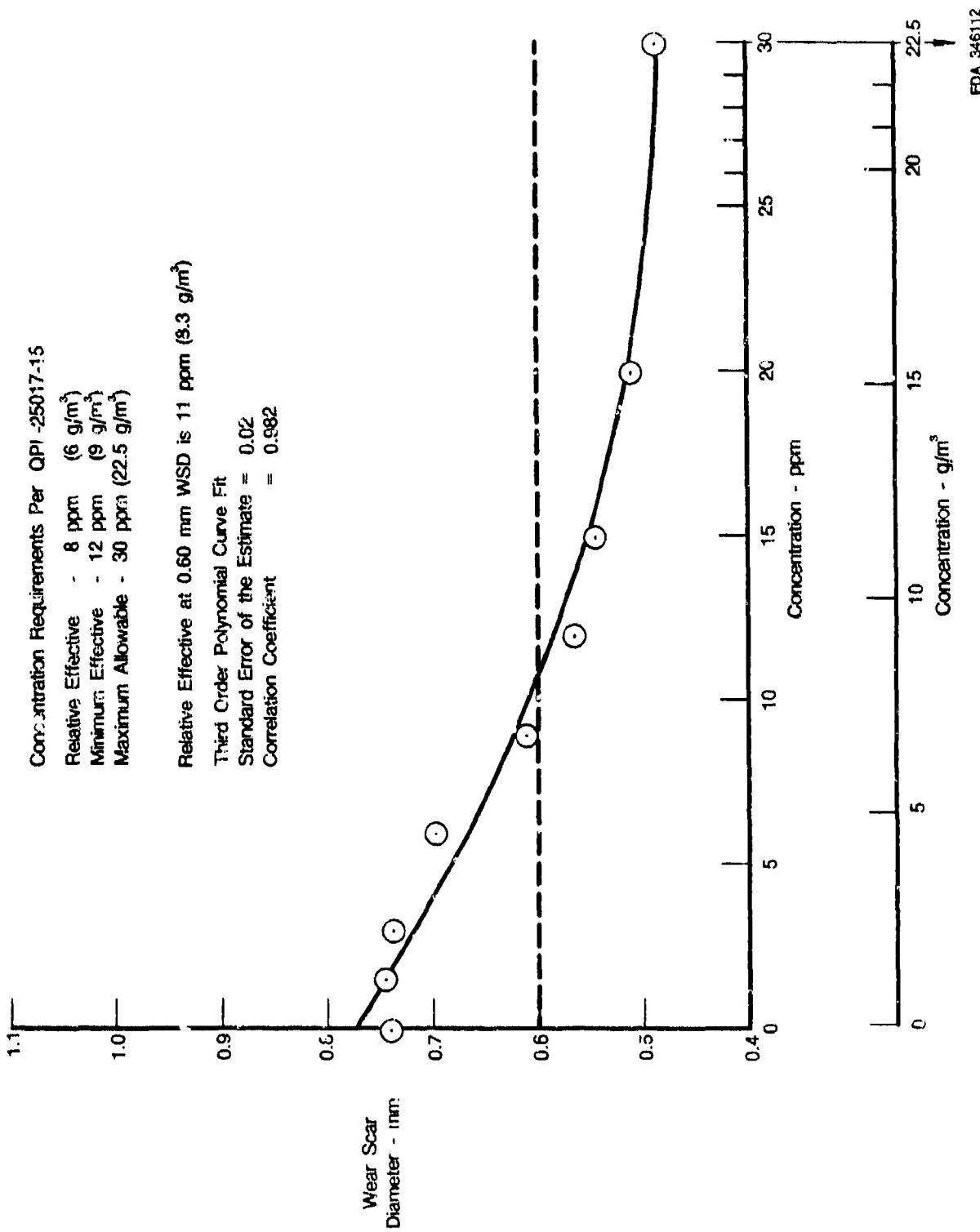
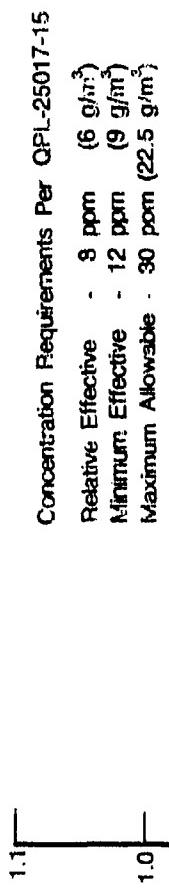


Figure D-10. — Effect of MOBILAD F-800 in ISOPAR-M



Relative Effective at 0.60 mm: WSD is 13 ppm (9.8 g/m³)

Second Order Polynomial Curve Fit
Standard Error of the Estimate = 0.022
Correlation Coefficient = 0.982

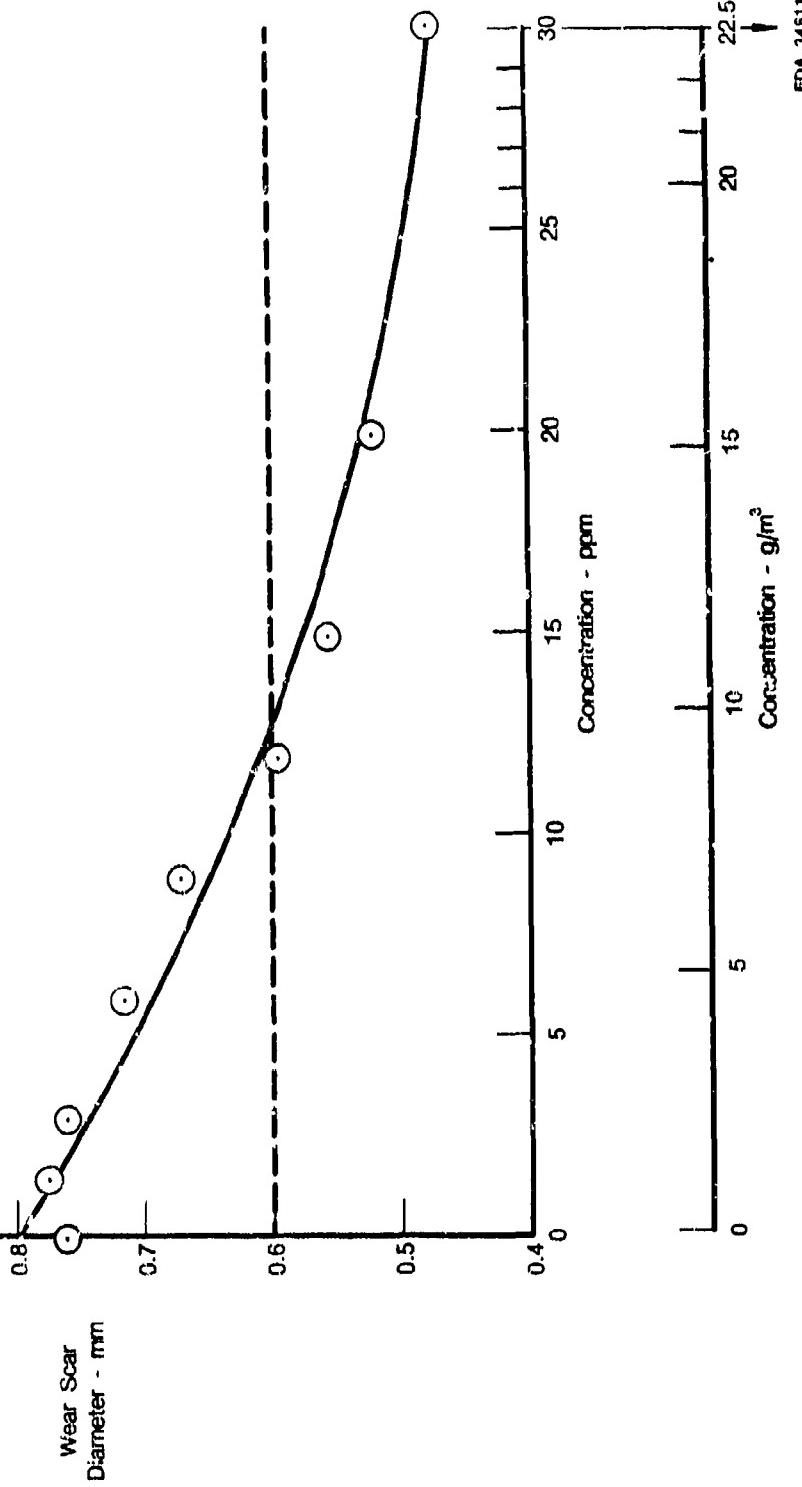


Figure D-11. — Effect of NALCO 5400 in ISOPAR-M

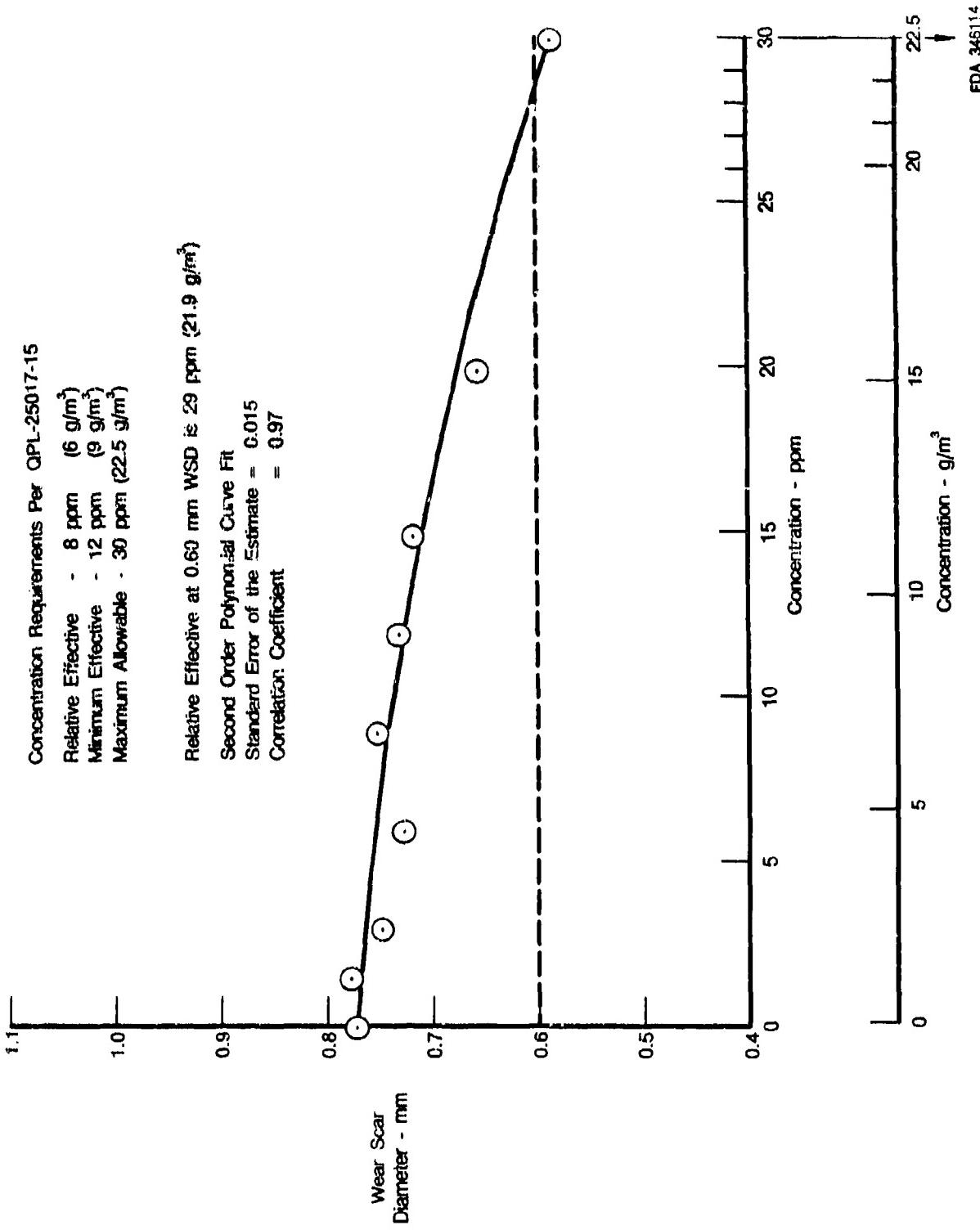
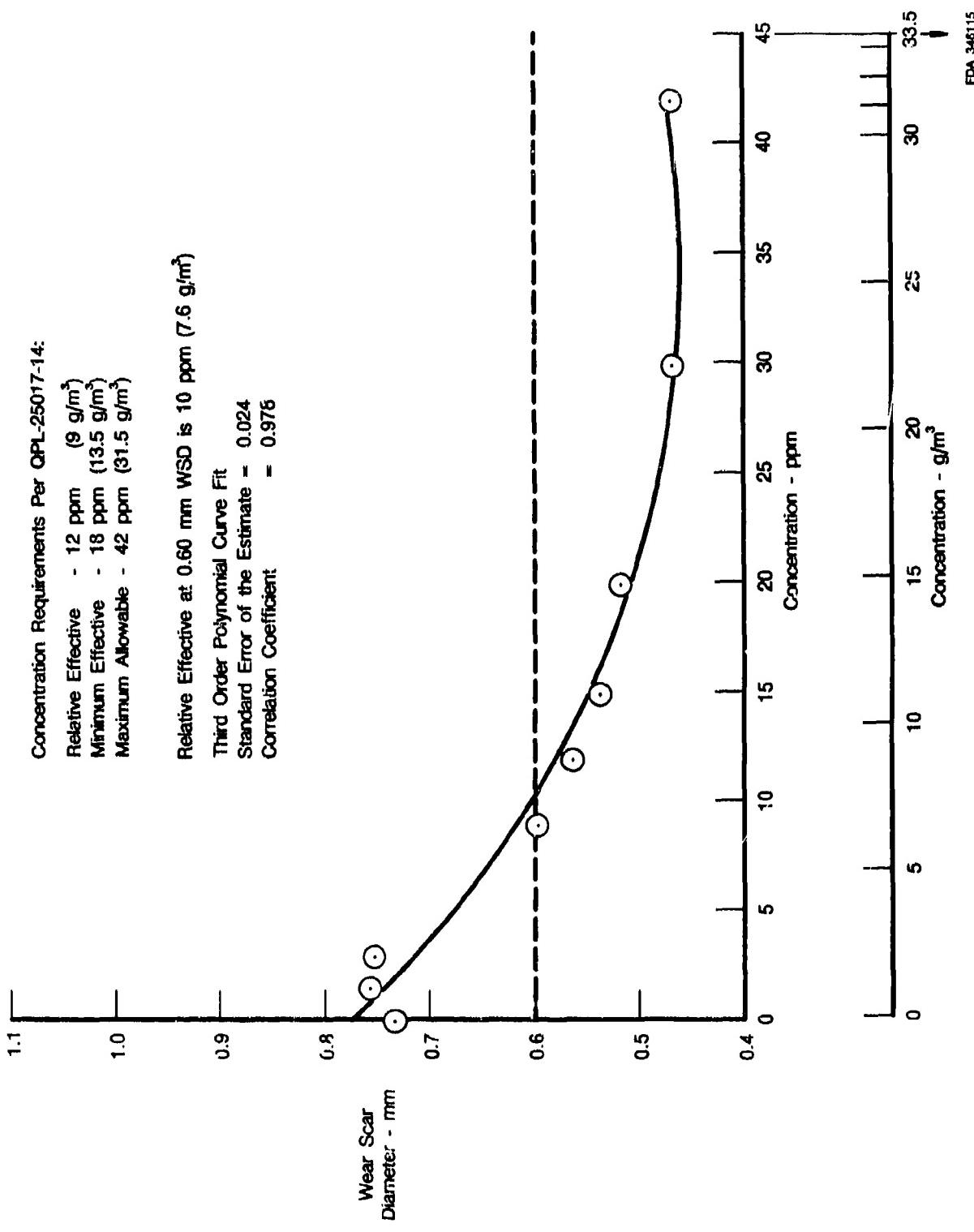


Figure D-12. — Effect of TOLAD 249 in ISOPAR-M



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Figure D-13. — Effect of P-3305 in ISOPAR-M

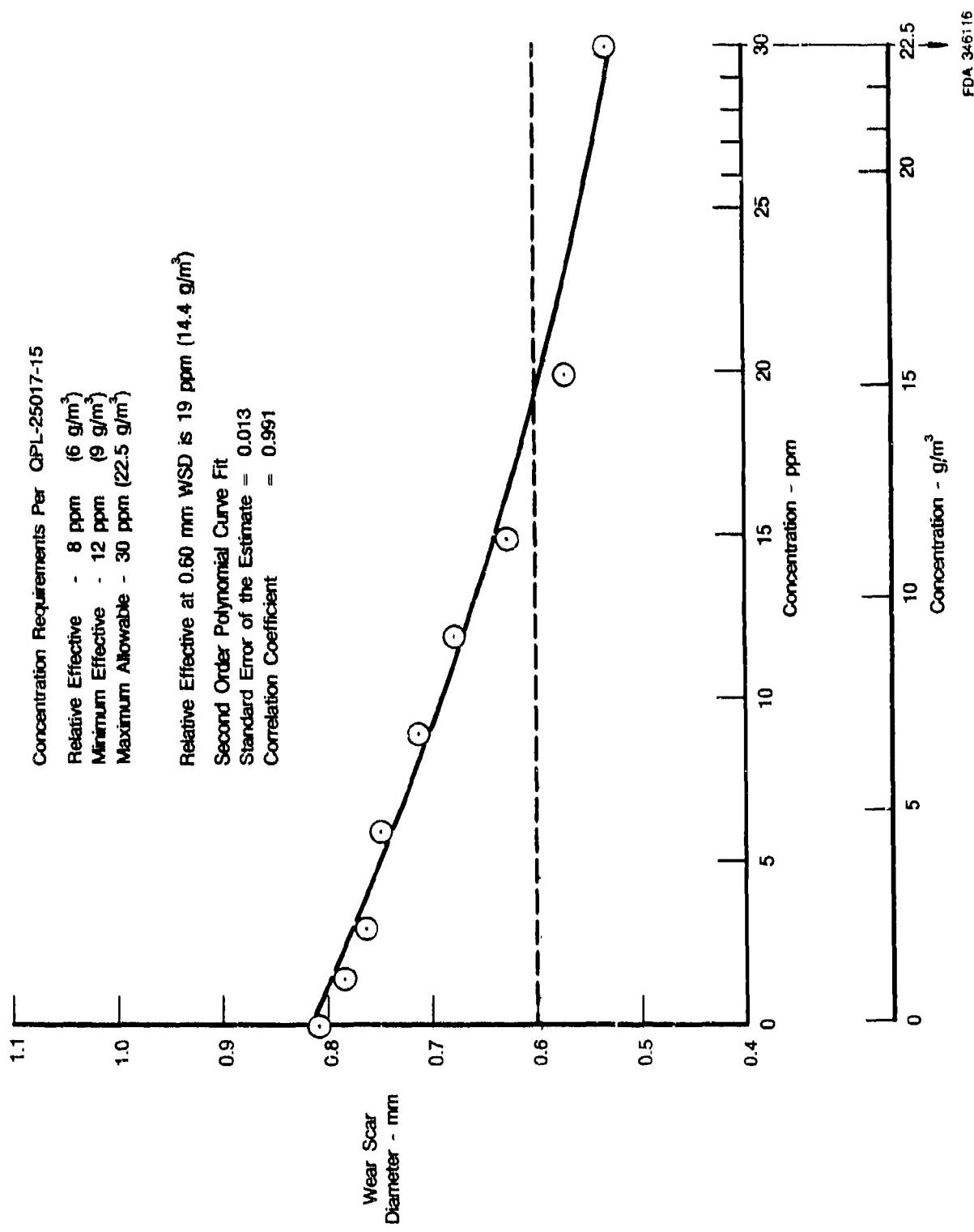


Figure D-14. — Effect of IPC-4445 in ISOPAR-M

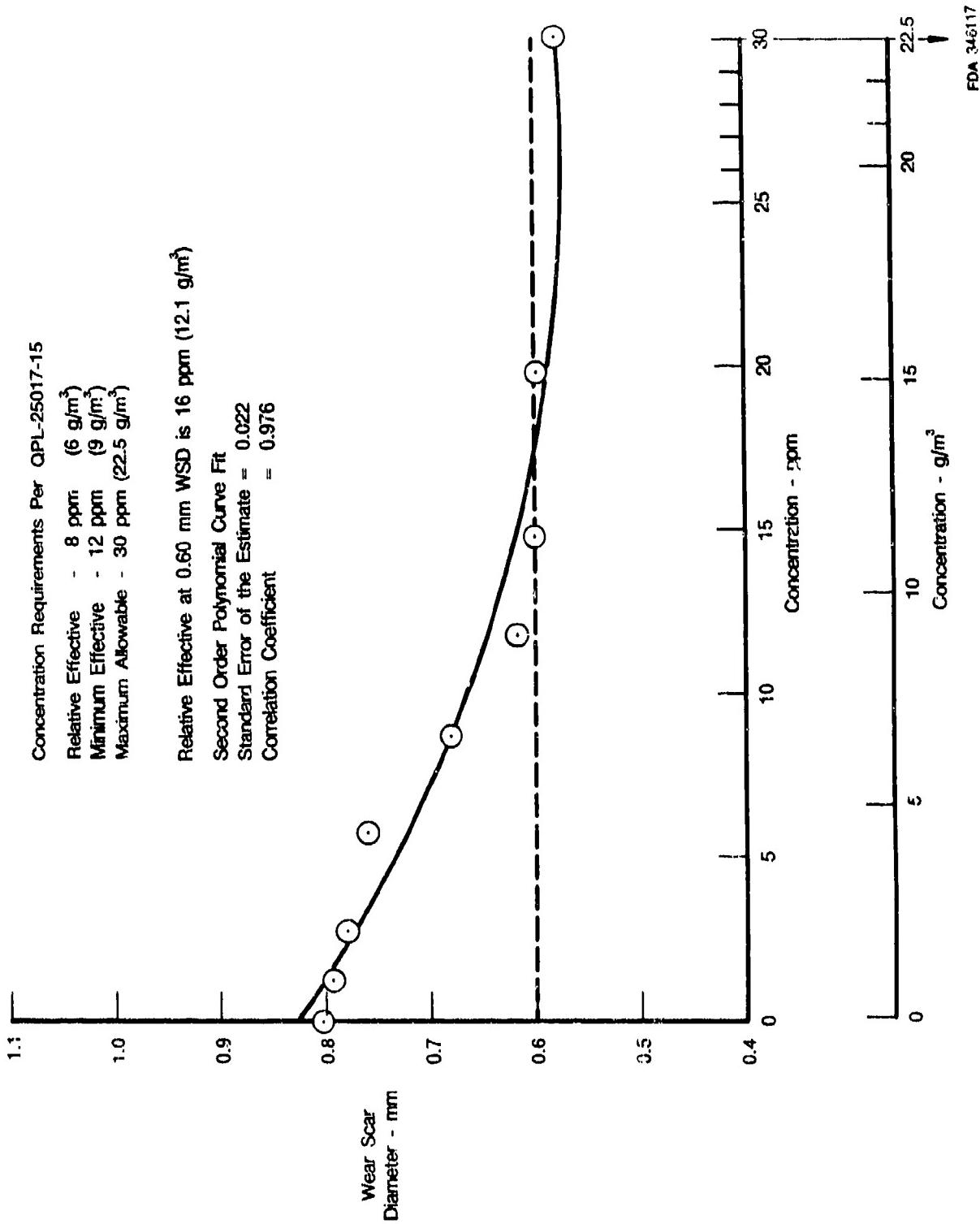


Figure D-15. — Effect of WELCHEM 91120 in ISOPAR-M

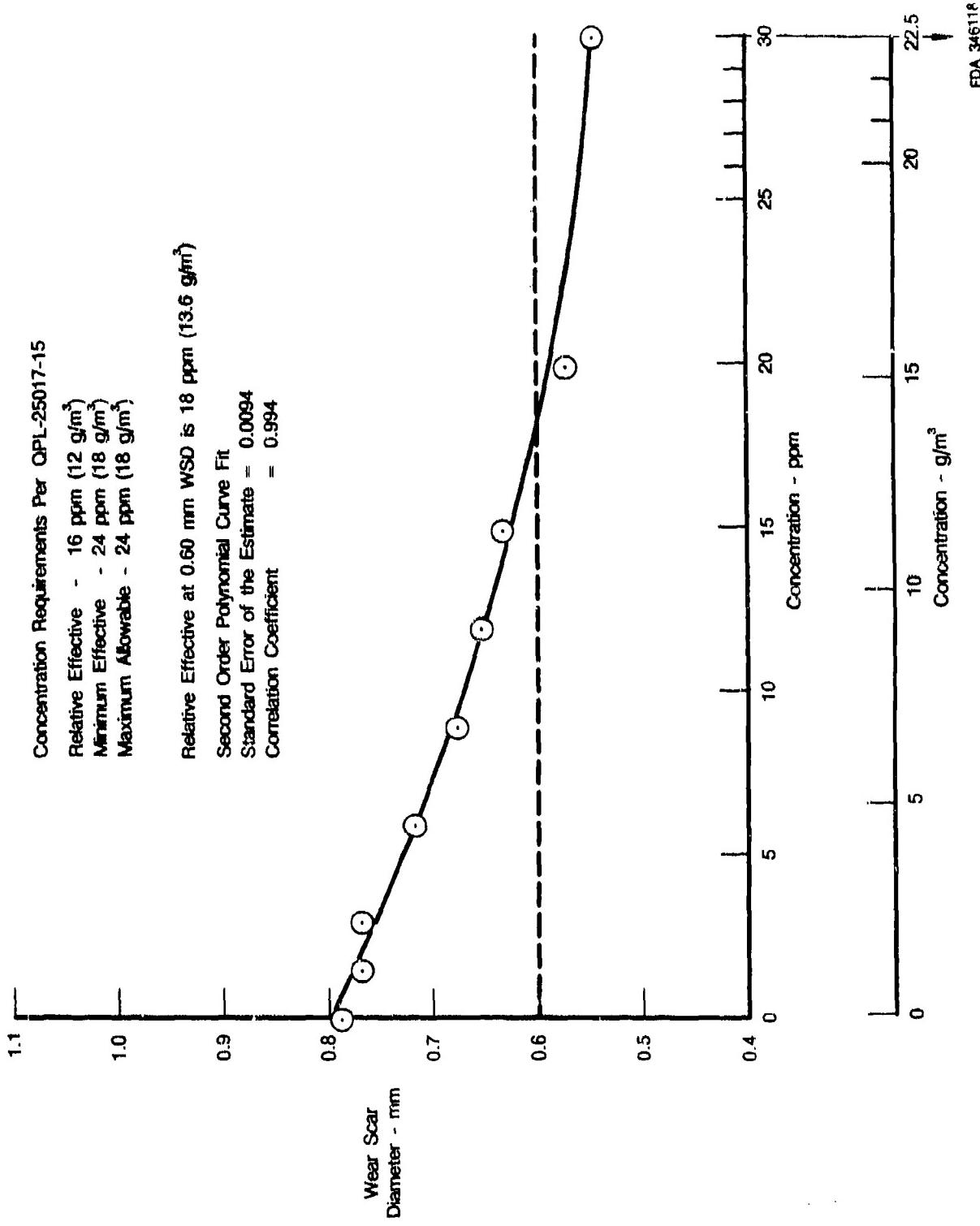


Figure D-16. — Effect of NUCHEM PCI-105 in ISOPAR-M

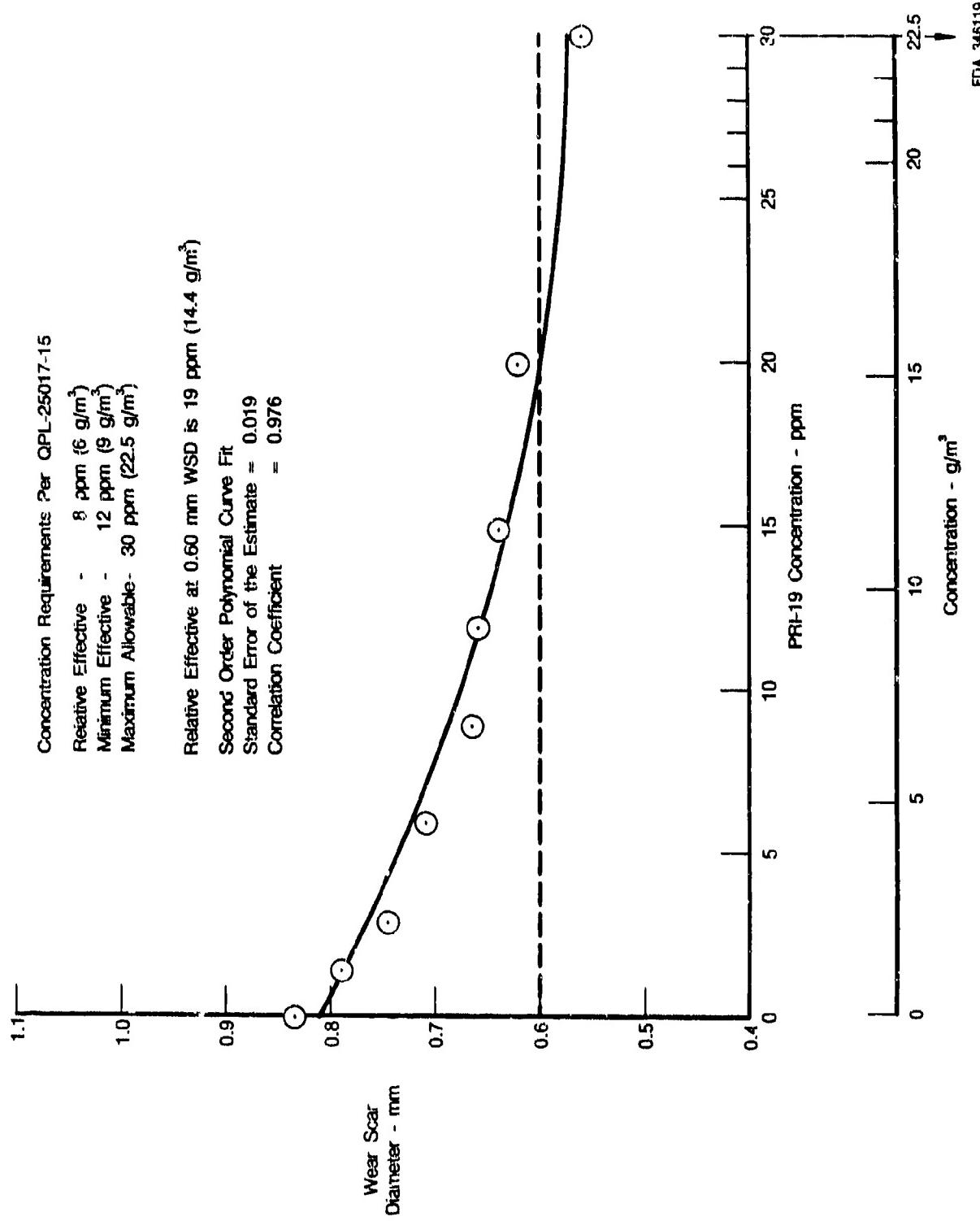


Figure D-17. — Effect of Apollo PRI-19 in Clay Treated JP-4

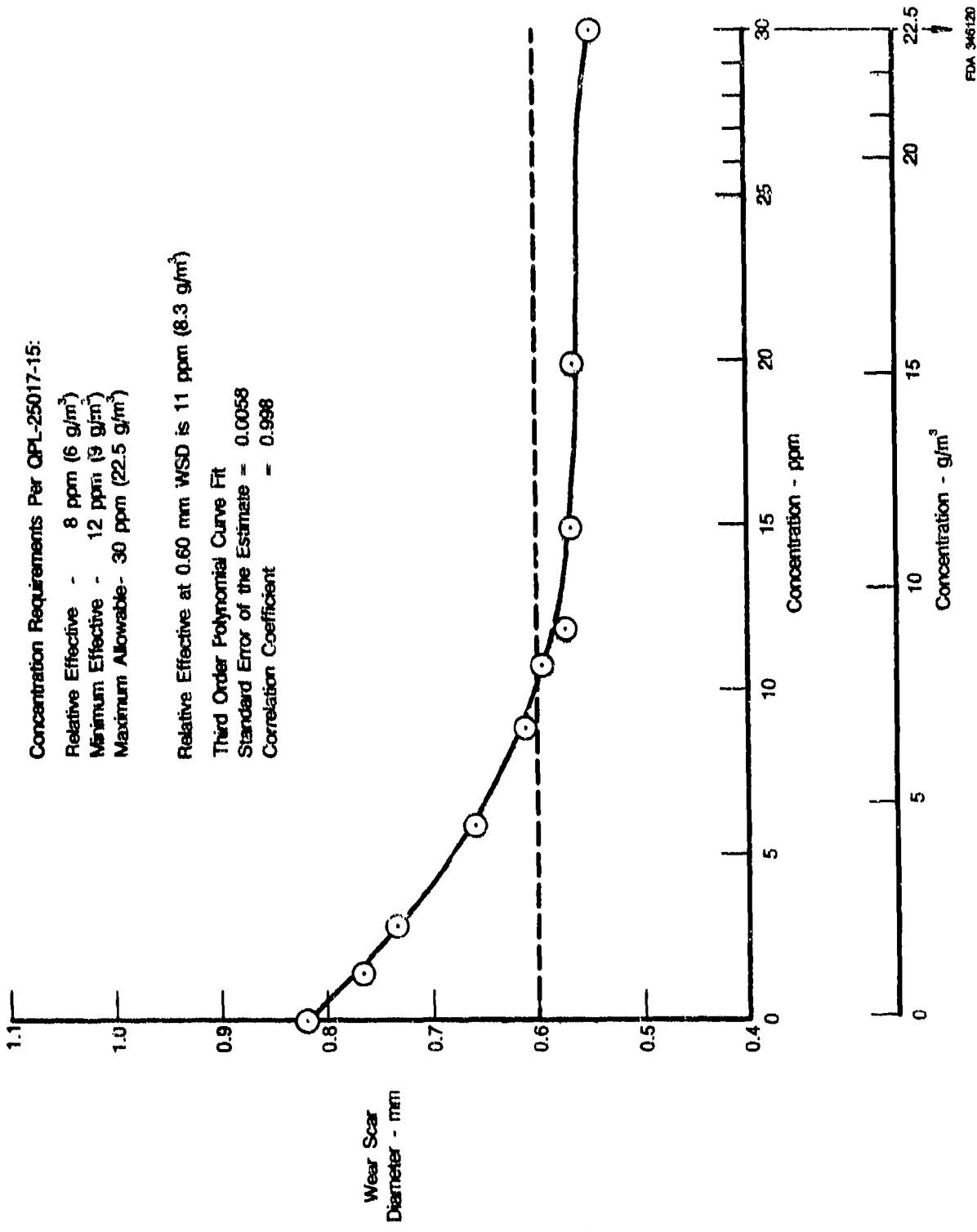
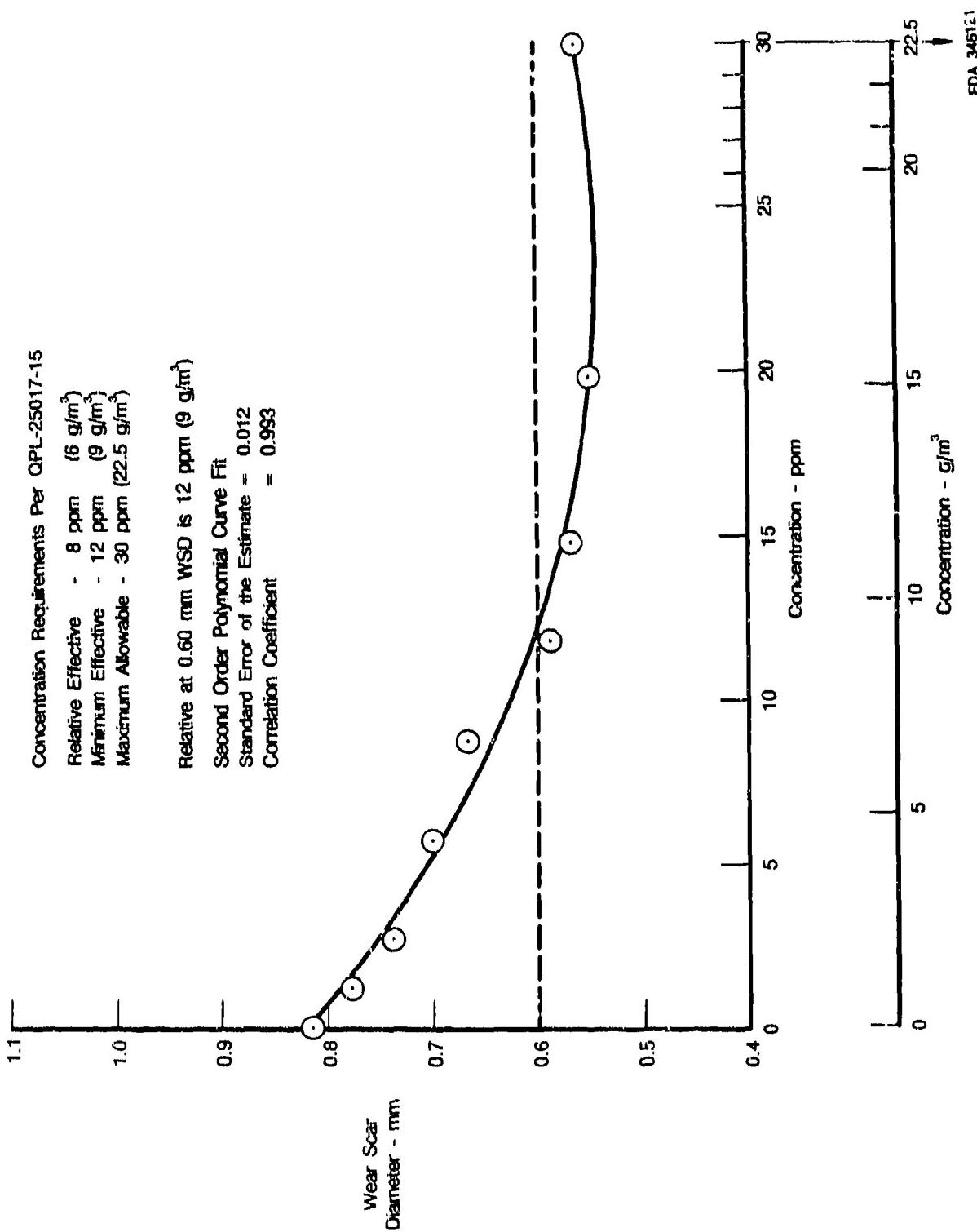


Figure D-18. — Effect of HITEC E-580 in Clay Treated JP-4



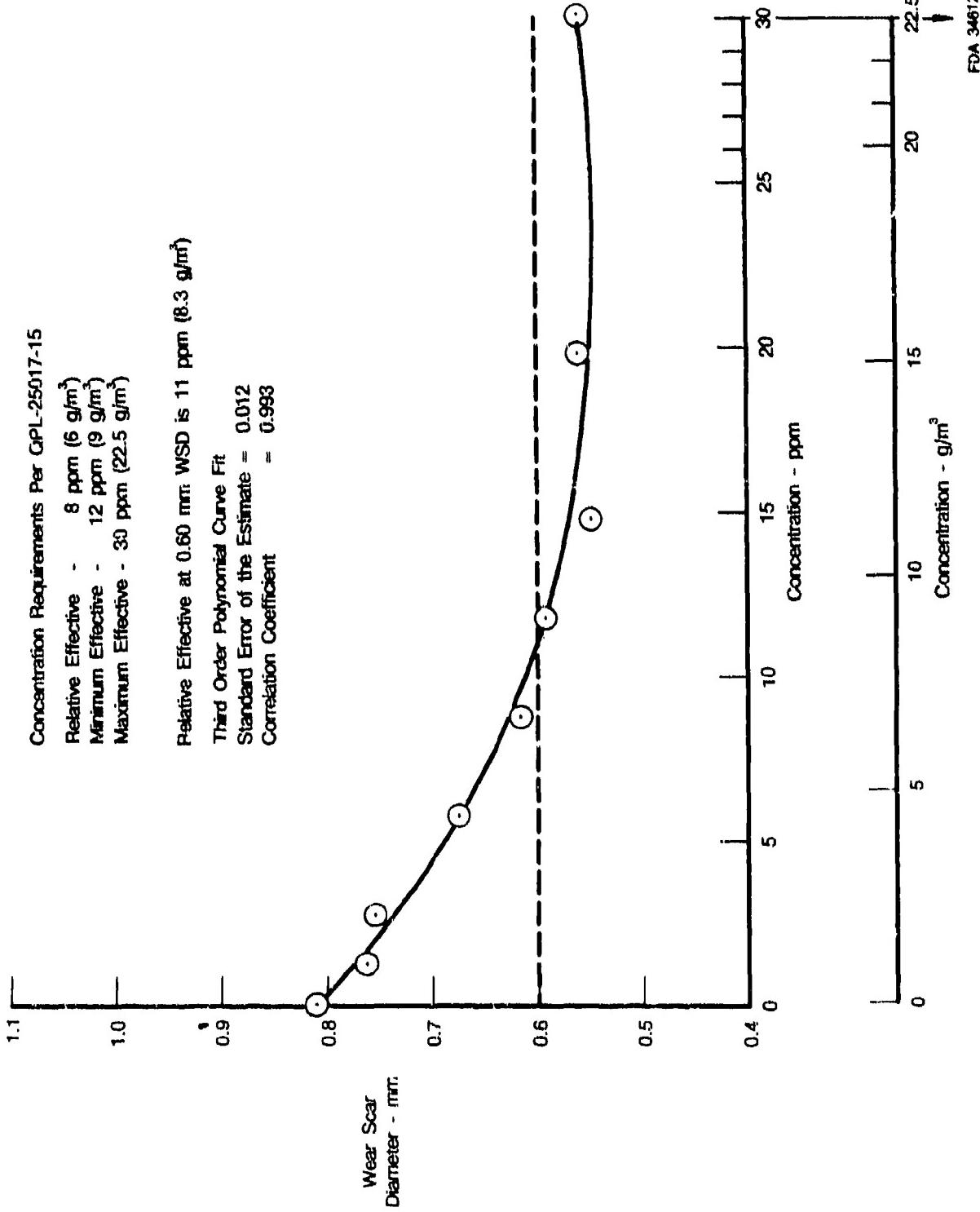
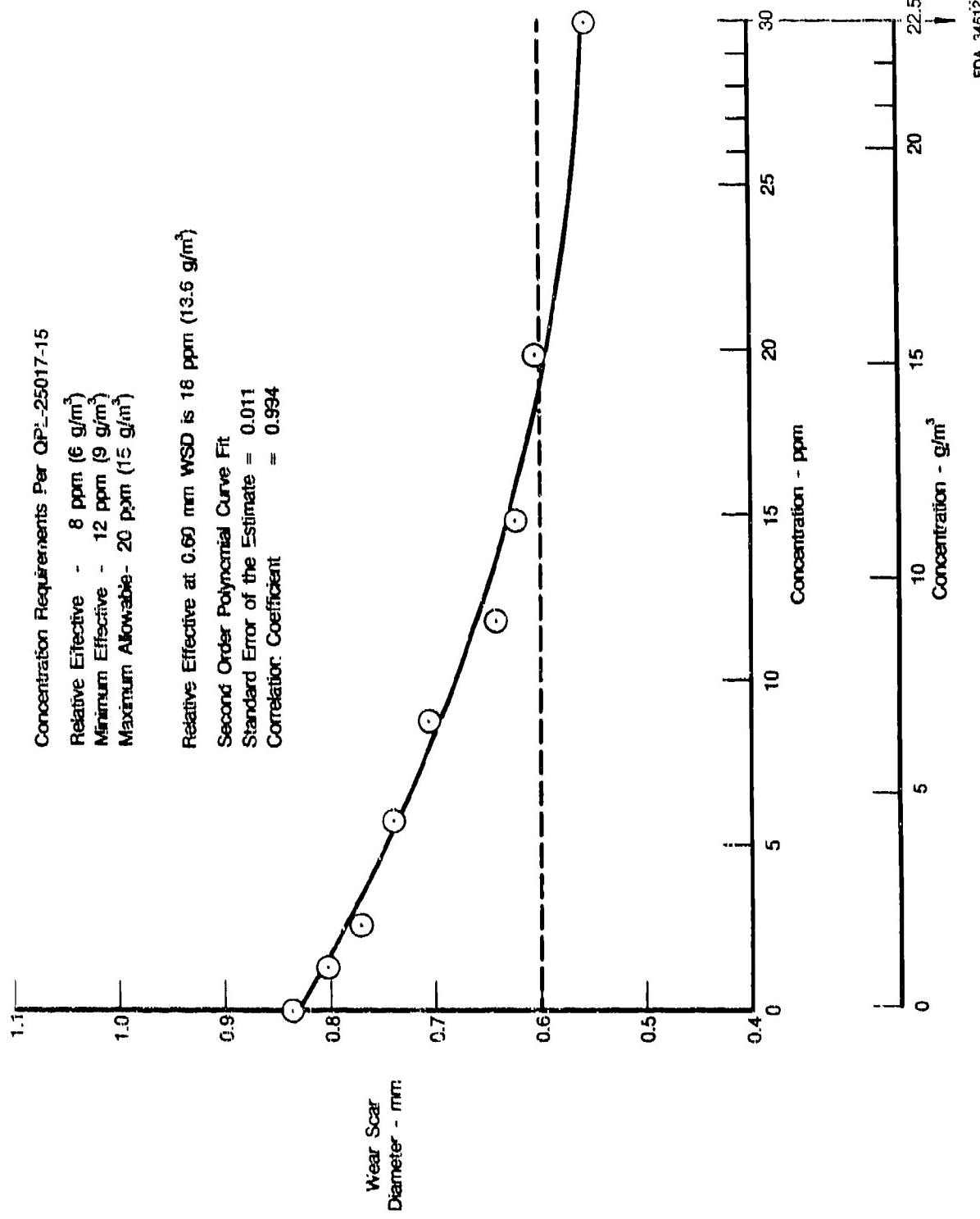


Figure D-20. — Effect of DCI-6A in Clay Treated JP-4



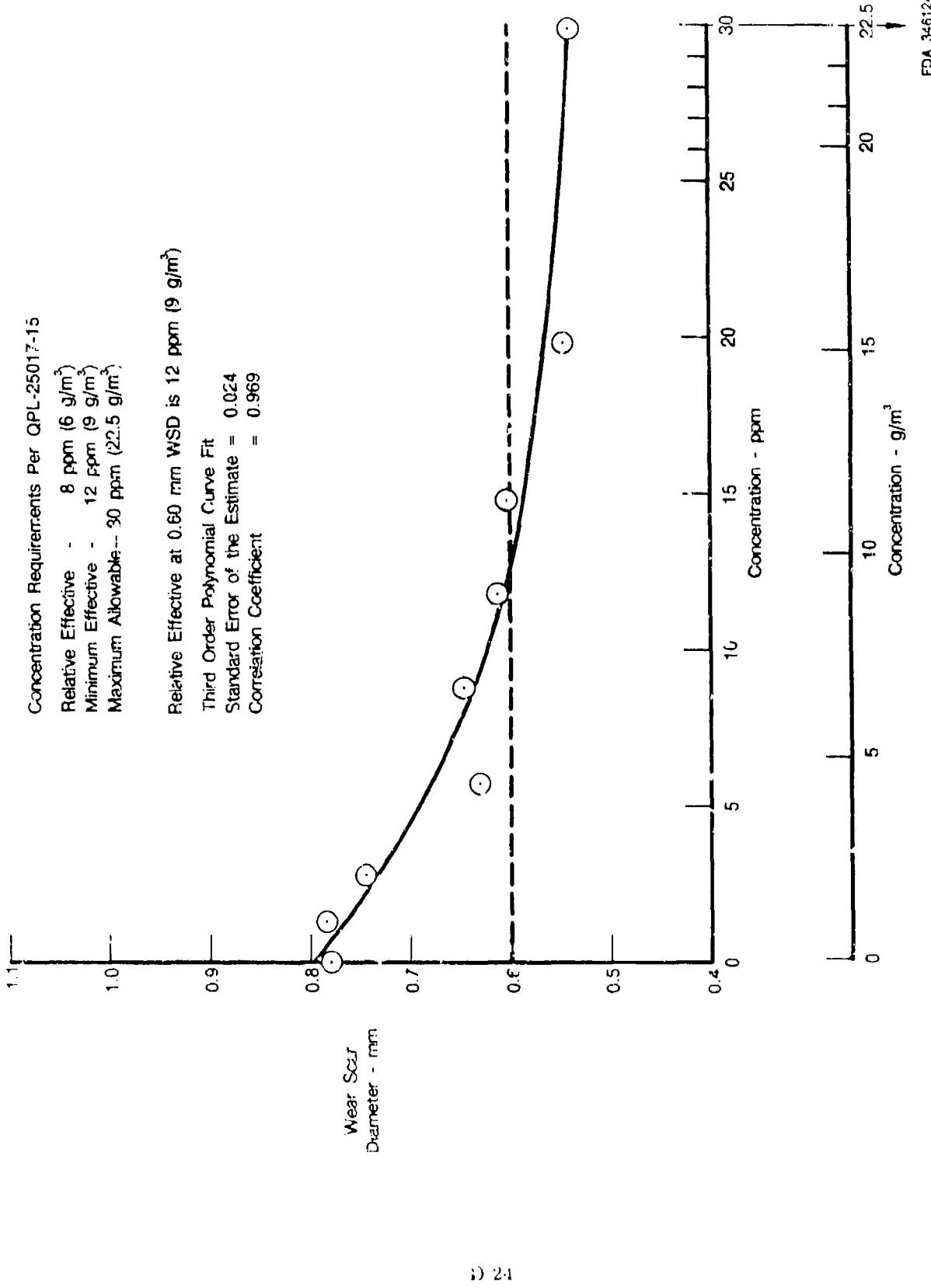


Figure D-22. — Effect of NALCO 5403 in Clay Treated JP-4

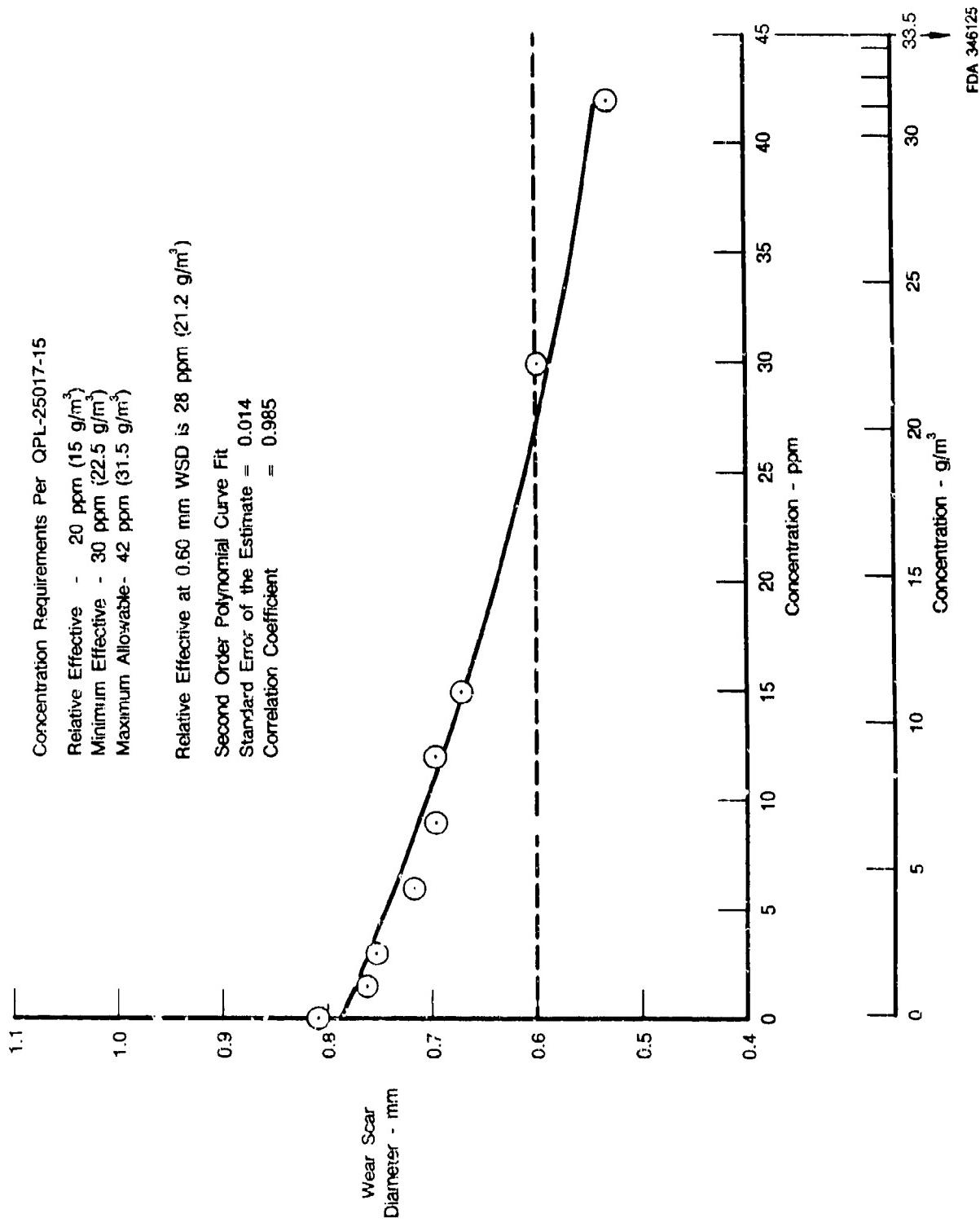


Figure D-23. — Effect of TOLAD 245 in City Treated JP-4

Concentration Requirements Per QPL-25017-15

Relative Effective - 8 ppm (6 g/m³)
Minimum Effective - 12 ppm (9 g/m³)
Maximum Allowable- 30 ppm (22.5 g/m³)

Relative Effective at 0.60 mm WSD is 9 ppm (6.8 g/m³)

Third Order Polynomial Curve Fit

Standard Error of the Estimate = 0.015
Correlation Coefficient = 0.987

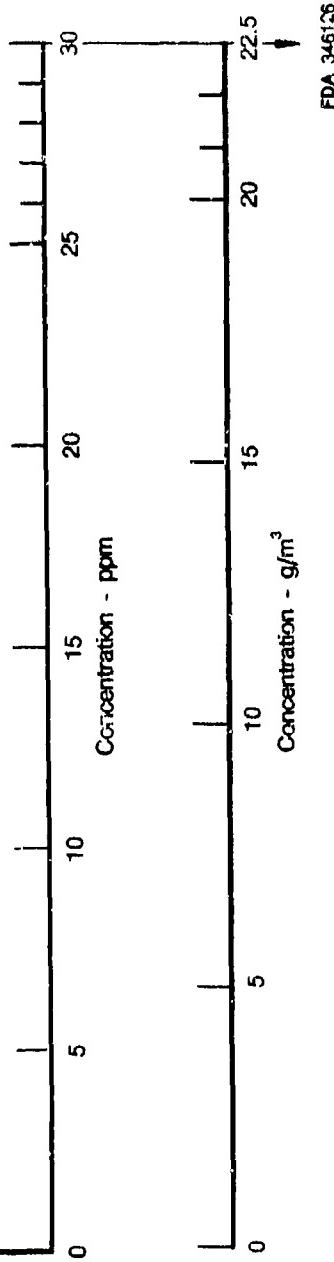
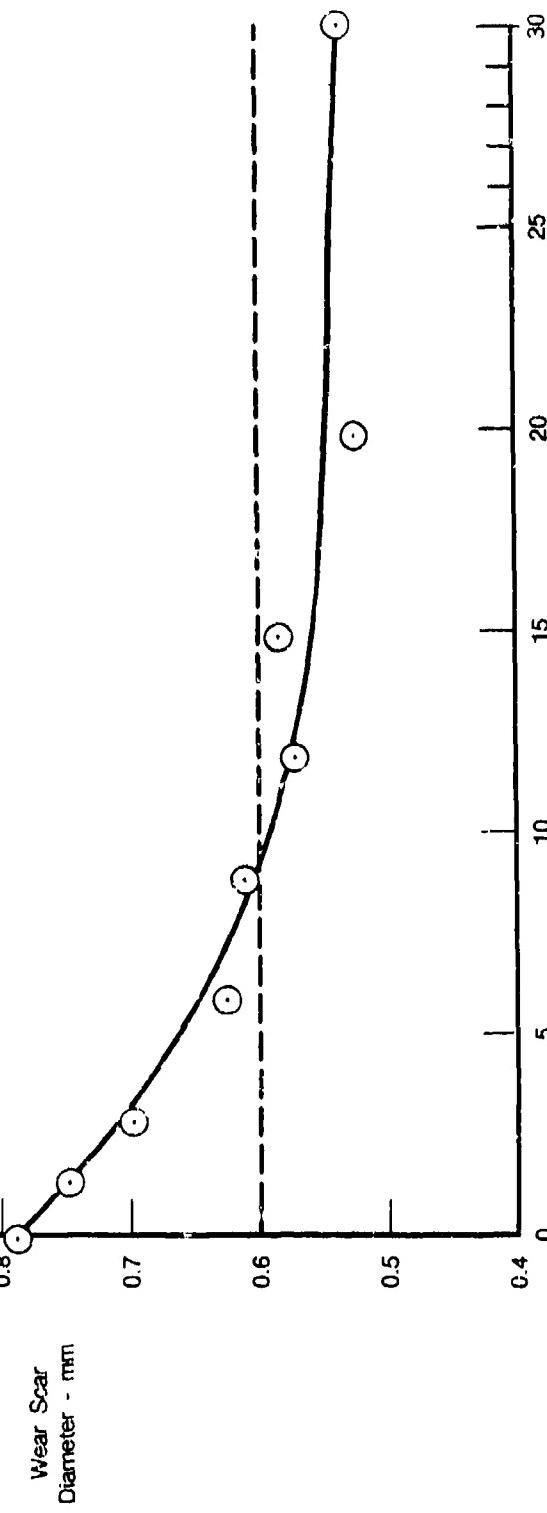


Figure D-24. — Effect of UNICOR-J in Clay Treated JP-4

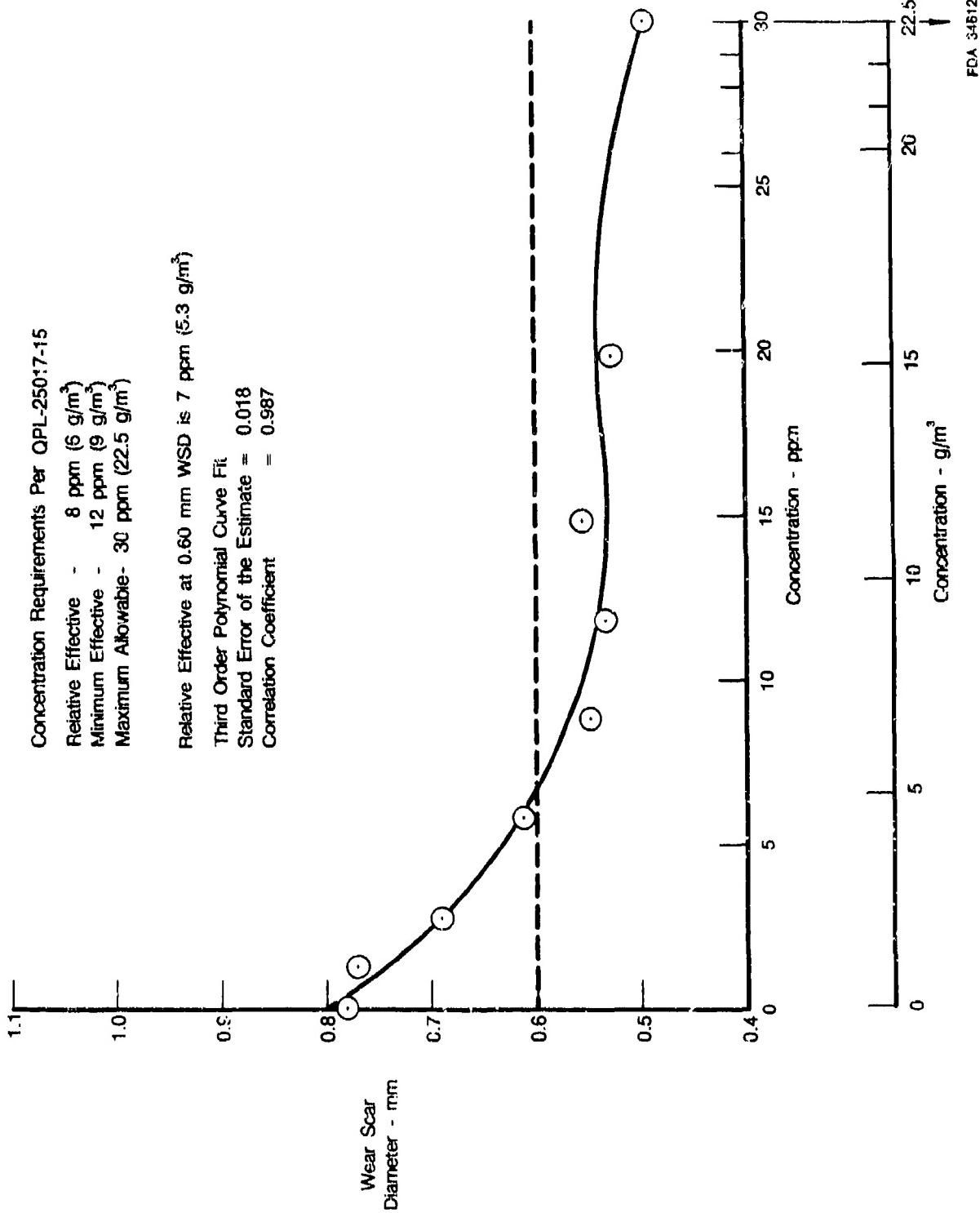


Figure D-25. — Effect of IPC-4410 in Clay Treated JP-4

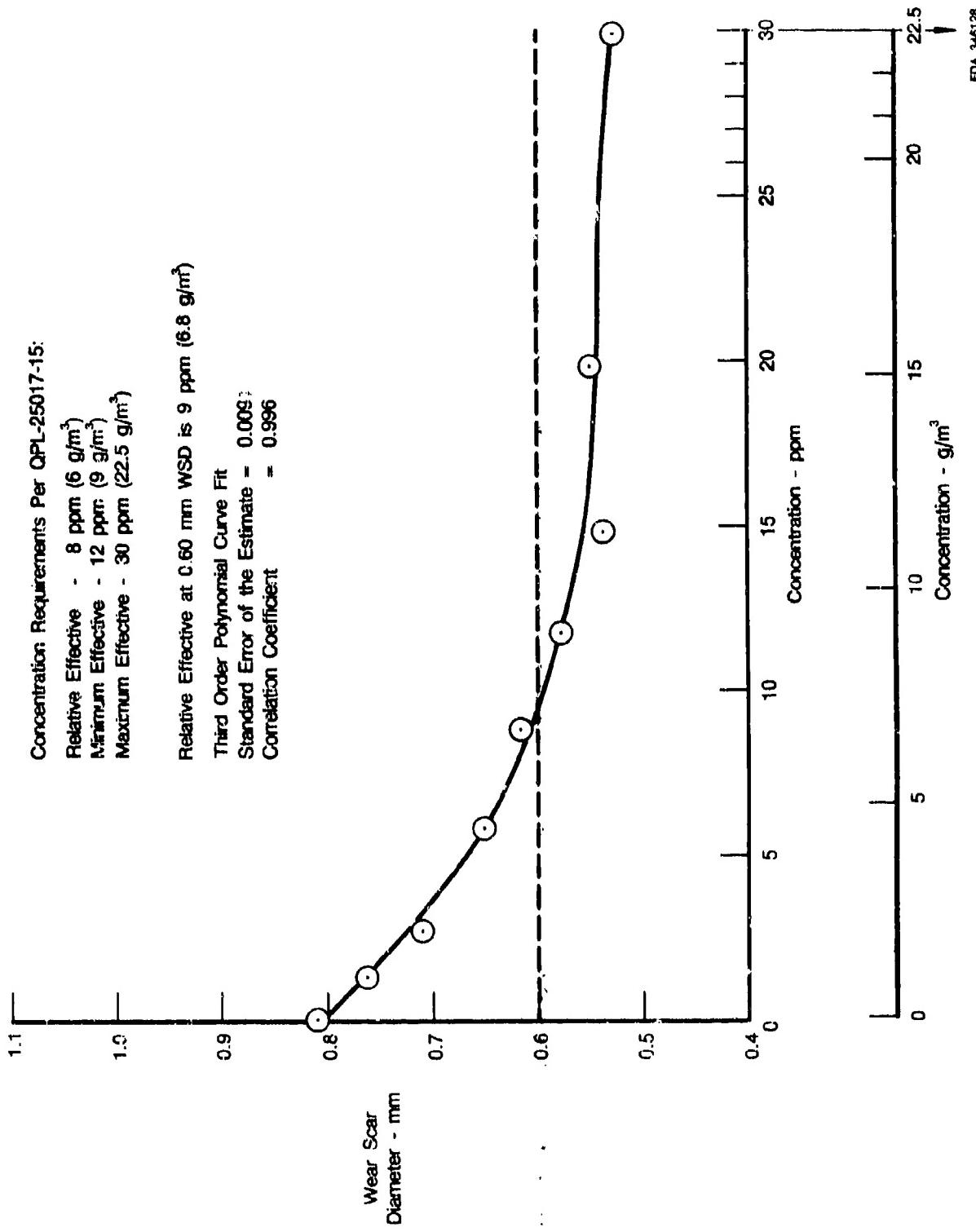


Figure D-26. — Effect of MOBILAD F-800 in Clay Treated JP-4

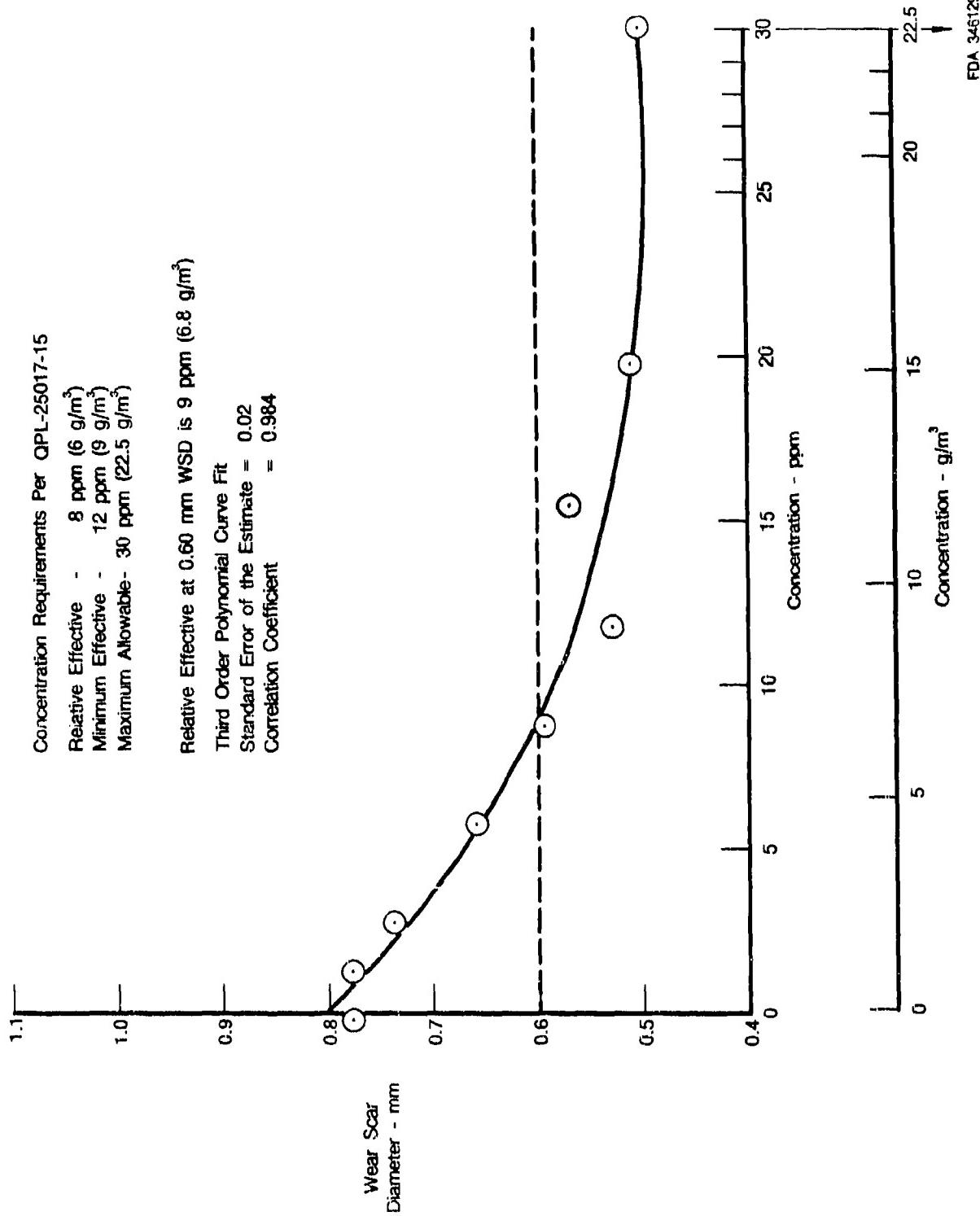


Figure D-27. — Effect of NALCO-5405 in Clay Treated JP-4

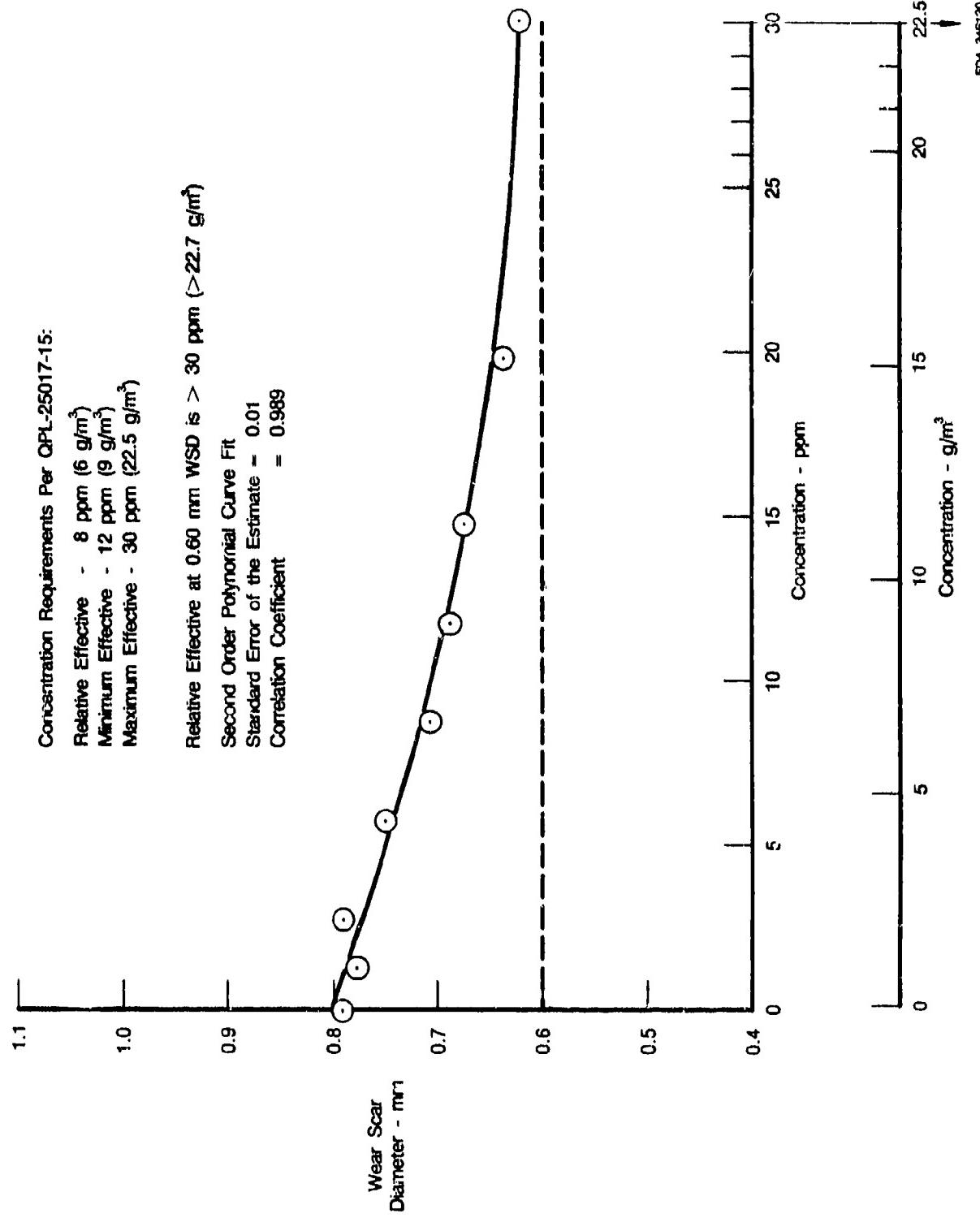


Figure D-28. — Effect of TOLAD 249 in Clay Treated JP-4

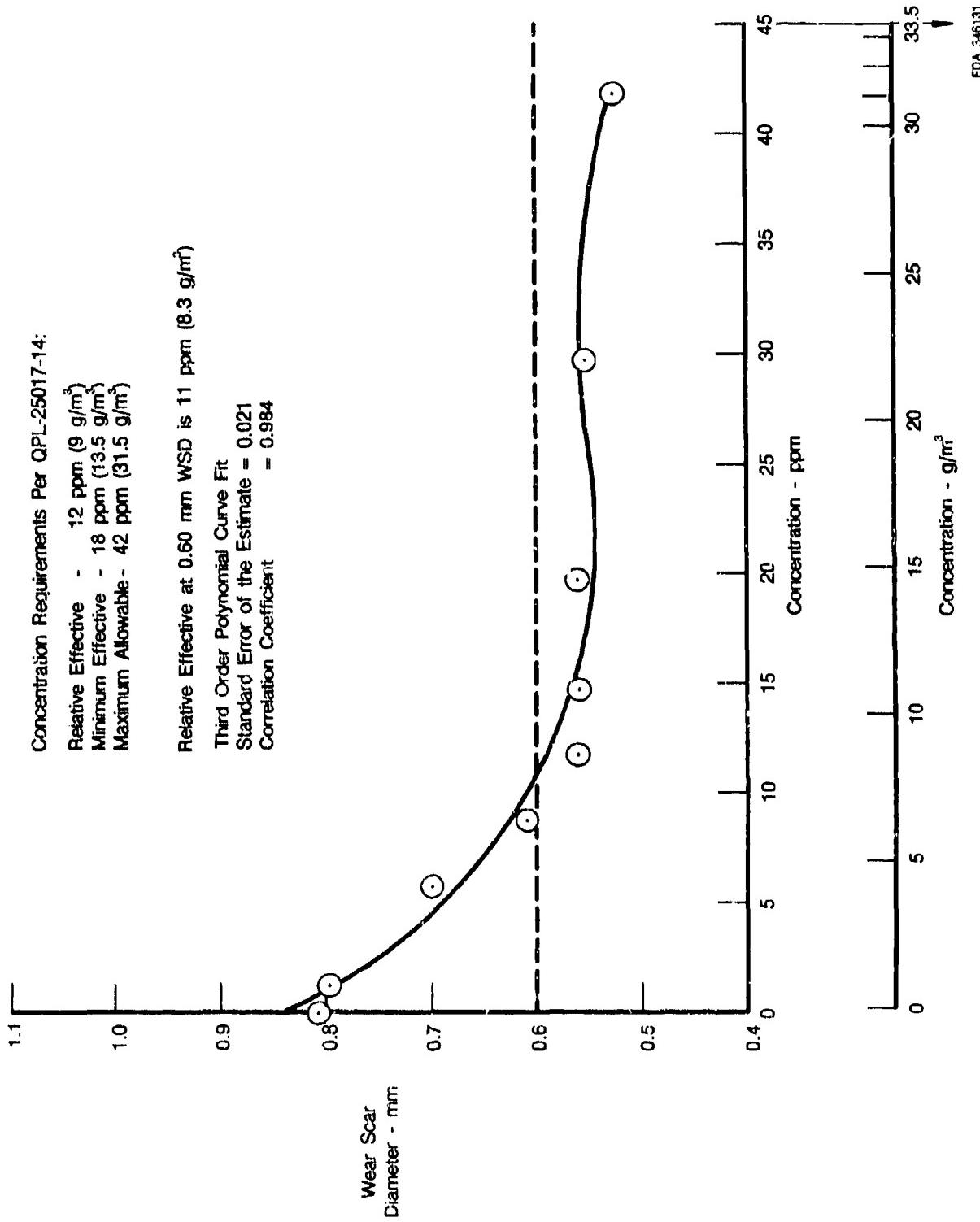


Figure D-29. — Effect of P-3305 in Clay Treated JP-4

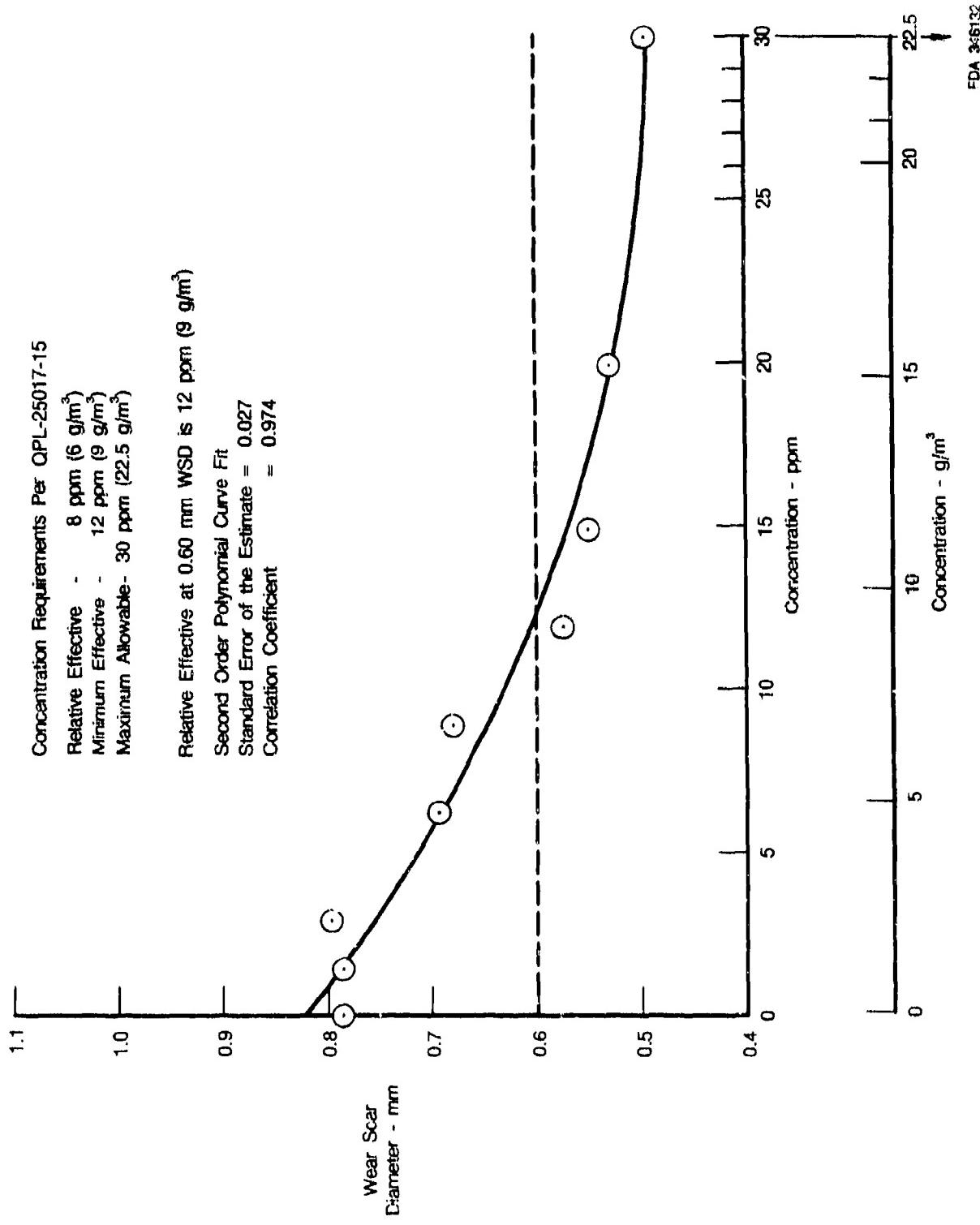


Figure D-50. — Effect of IPC-4445 in Clay Treated JP-4

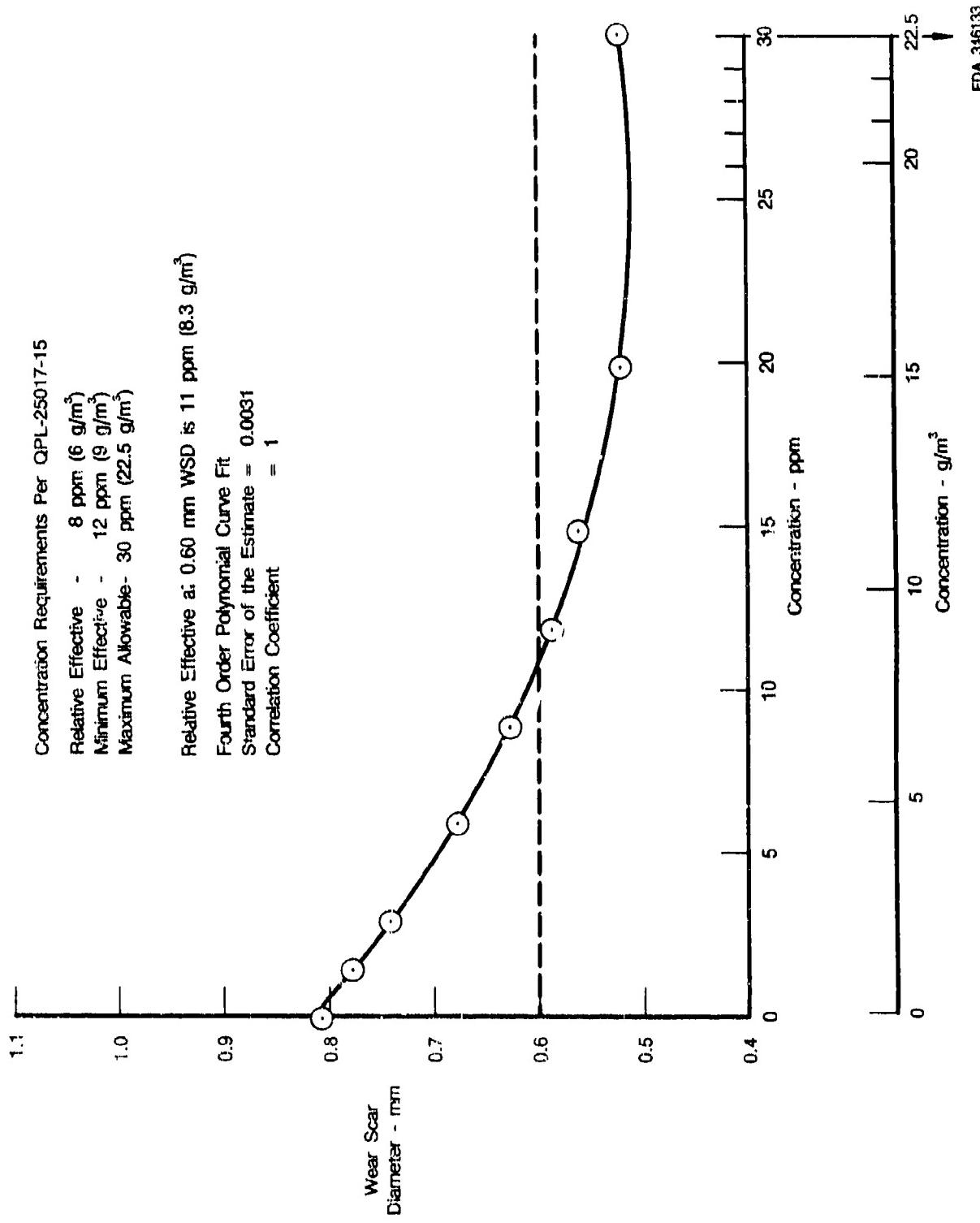


Figure D-31. — Effect of WELCHEM 9112C in Clay Treated JP-4

Concentration Requirements Per QPL-25017-15

Relative Effective - 16 ppm (12 g/m^3)
Minimum Effective - 24 ppm (18 g/m^3)
Maximum Allowable- 24 ppm (18 g/m^3)

Relative Effective at 0.60 mm WSD is 17 ppm (12.8 g/m^3)

Third Order Polynomial Curve Fit

Standard Error of the Estimate = 0.0081
Correlation Coefficient = 0.996

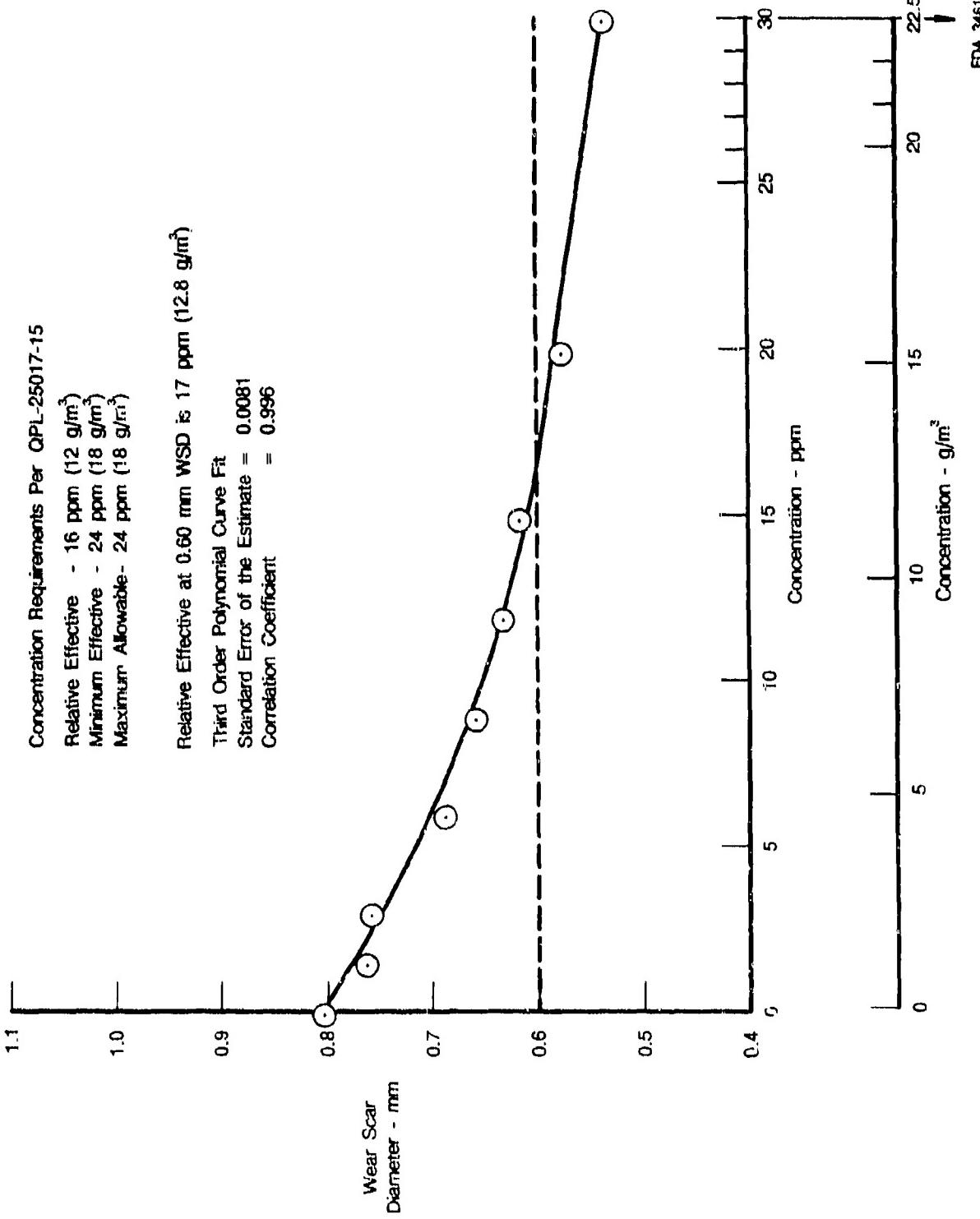


Figure D-32. — Effect of NUCHEM PCI-105 in Clay Treated JP-4

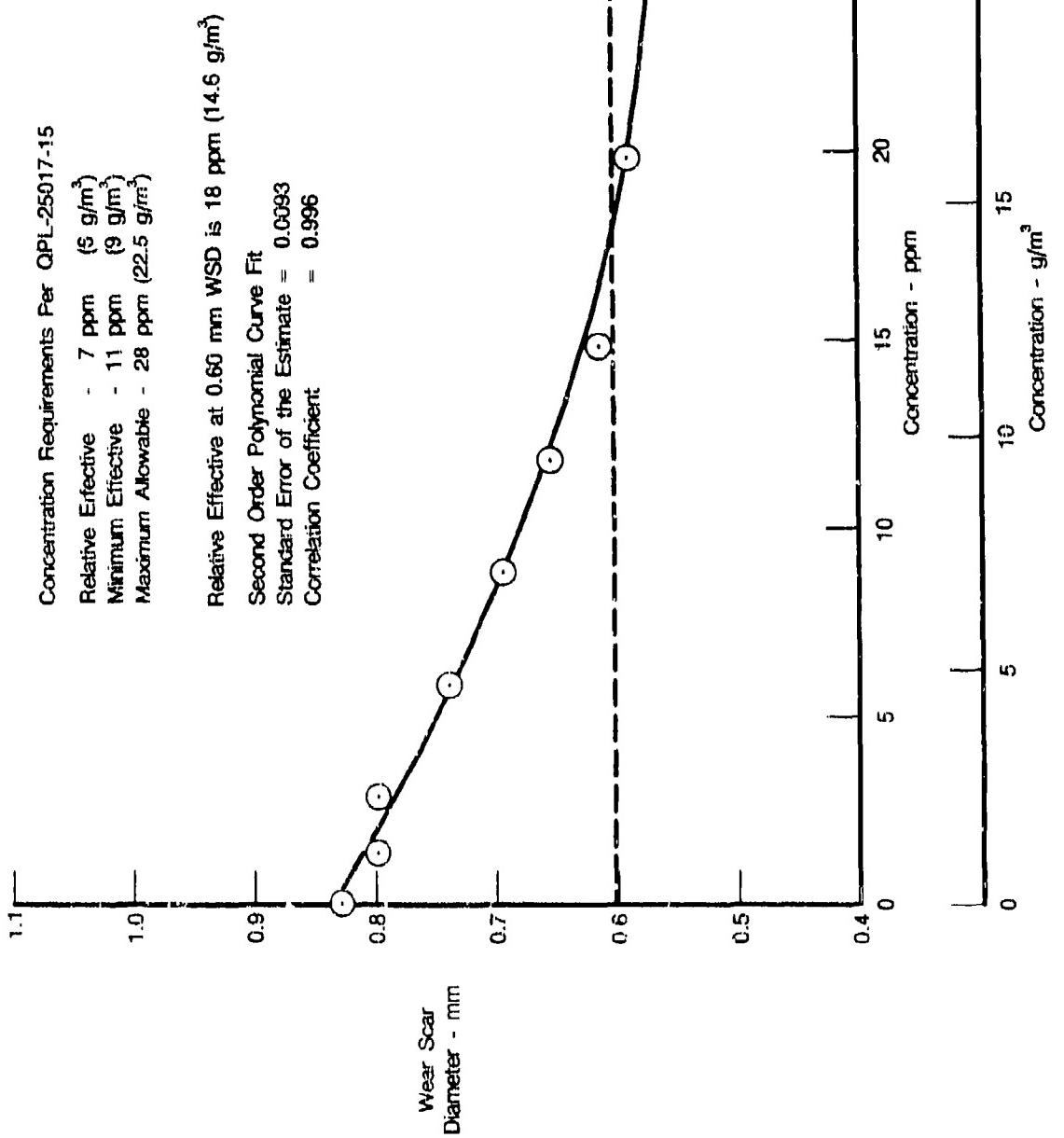


Figure D-33. — Effect of Apcito PRI-1E in Clay Treated JP-8

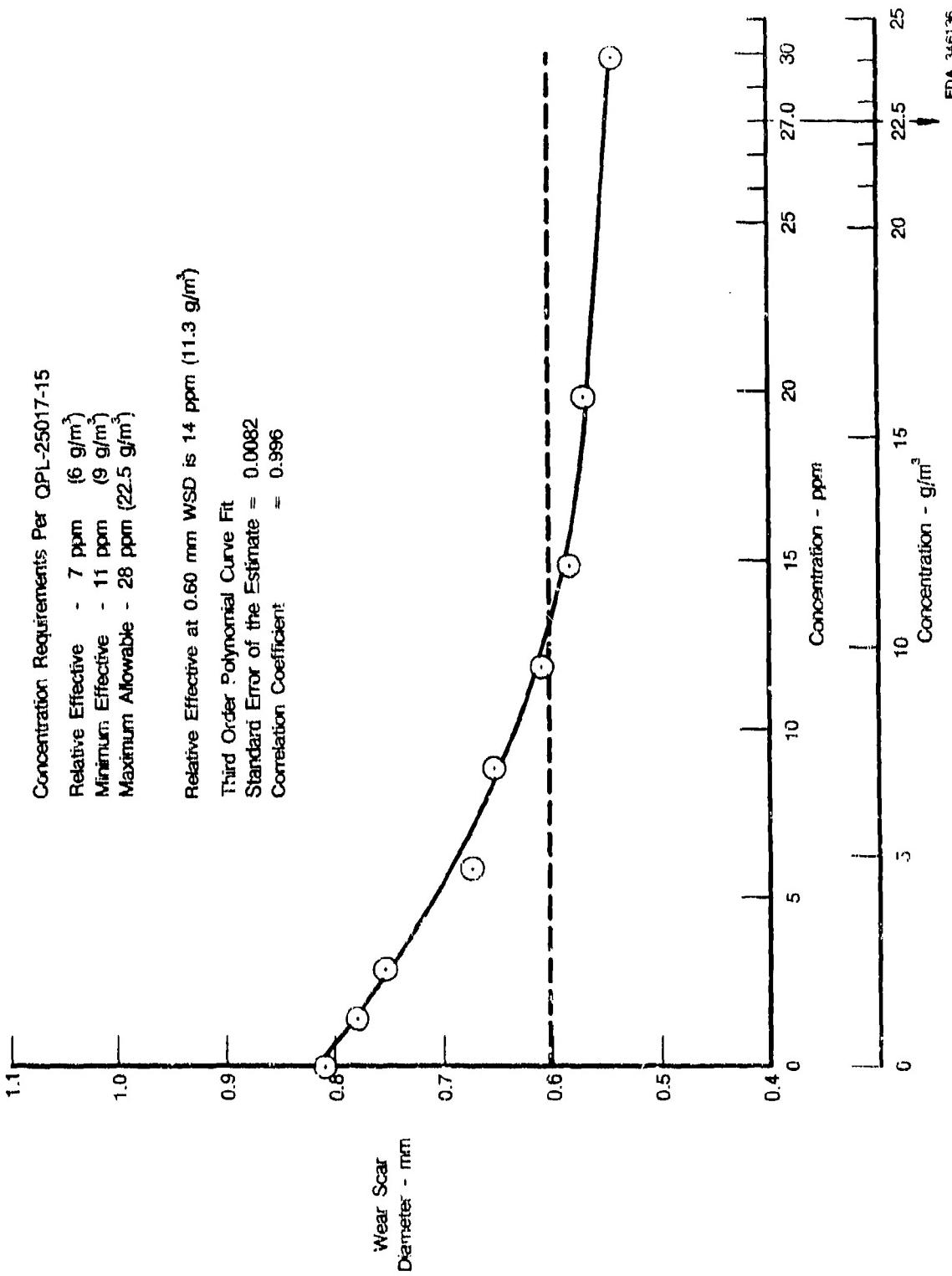


Figure D-34. — Effect of HITEC E-580 in Clay Treated JP-8

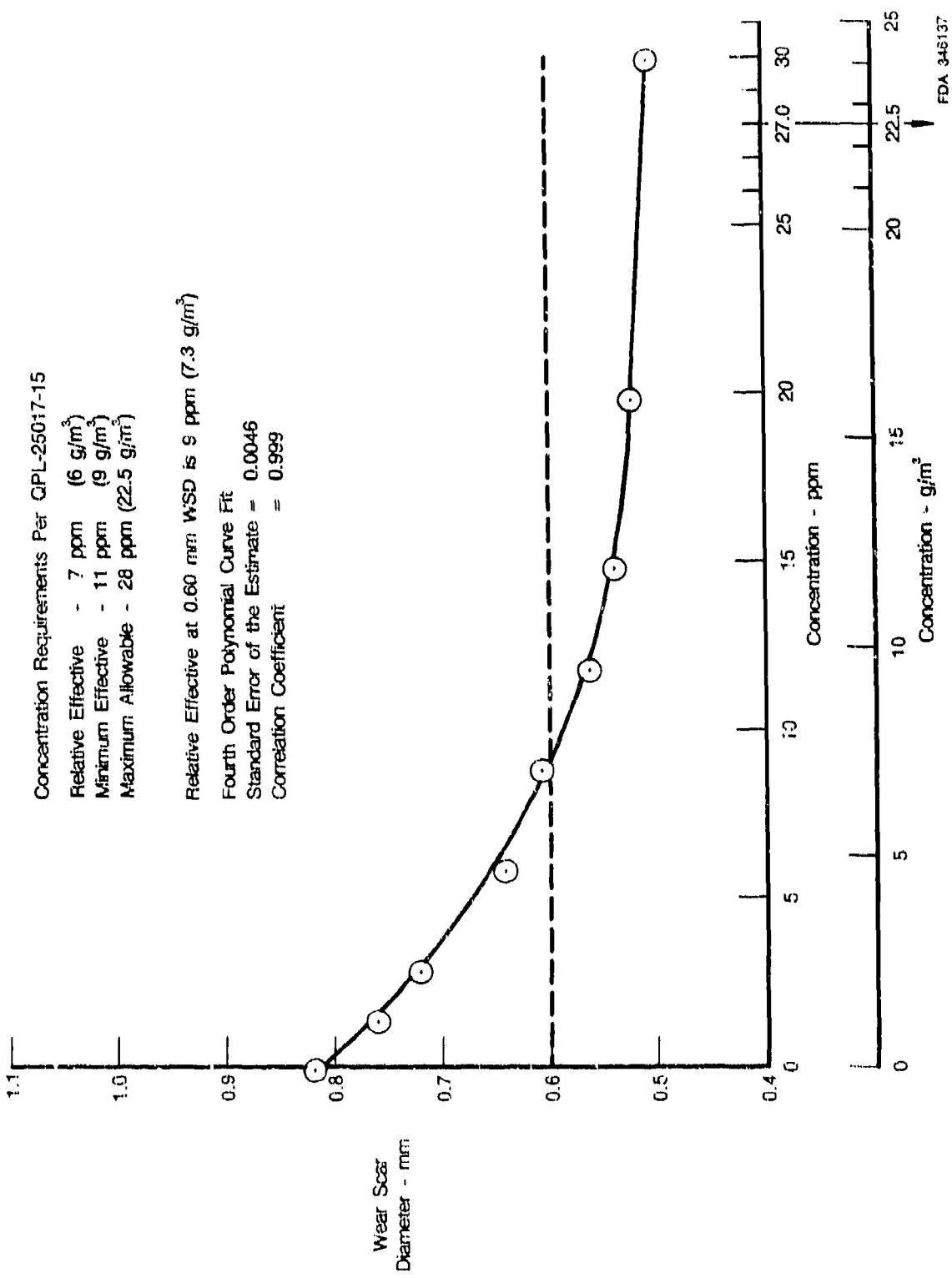


Figure D-35. — Effect of DCI-4A in Clay Treated JP-8

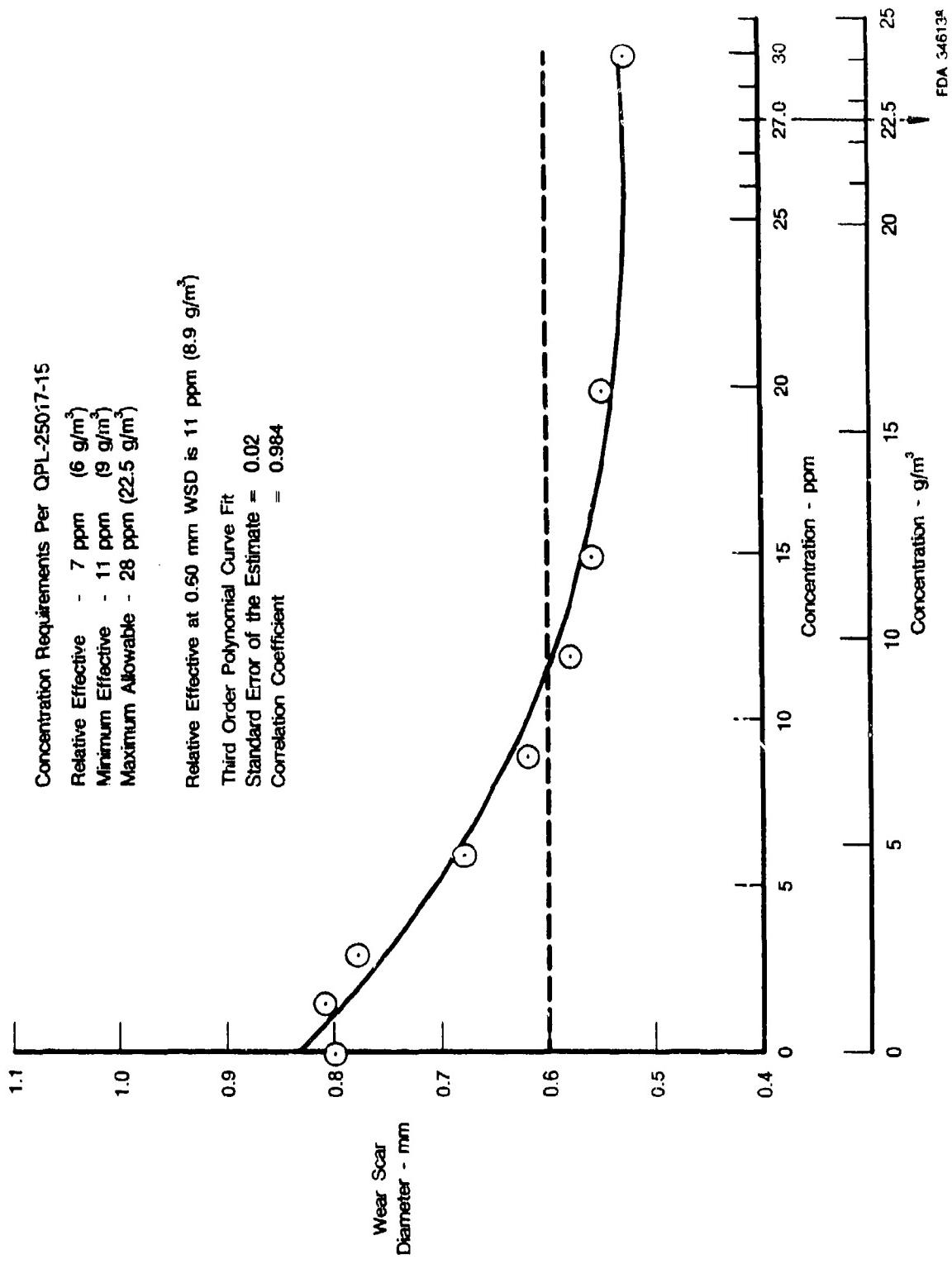


Figure D-36. — Effect of DCI-6A in Clay Treated JP-8

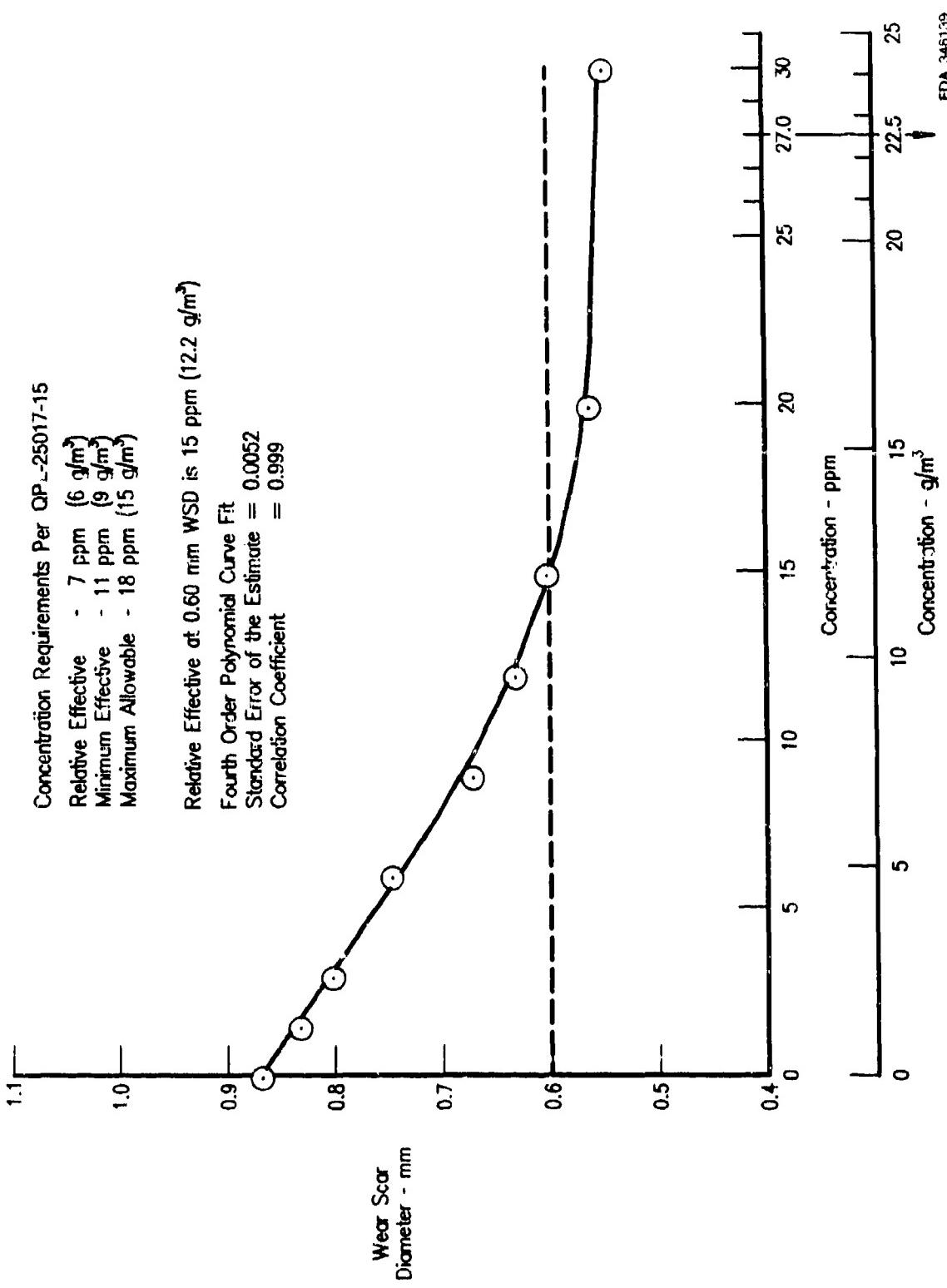
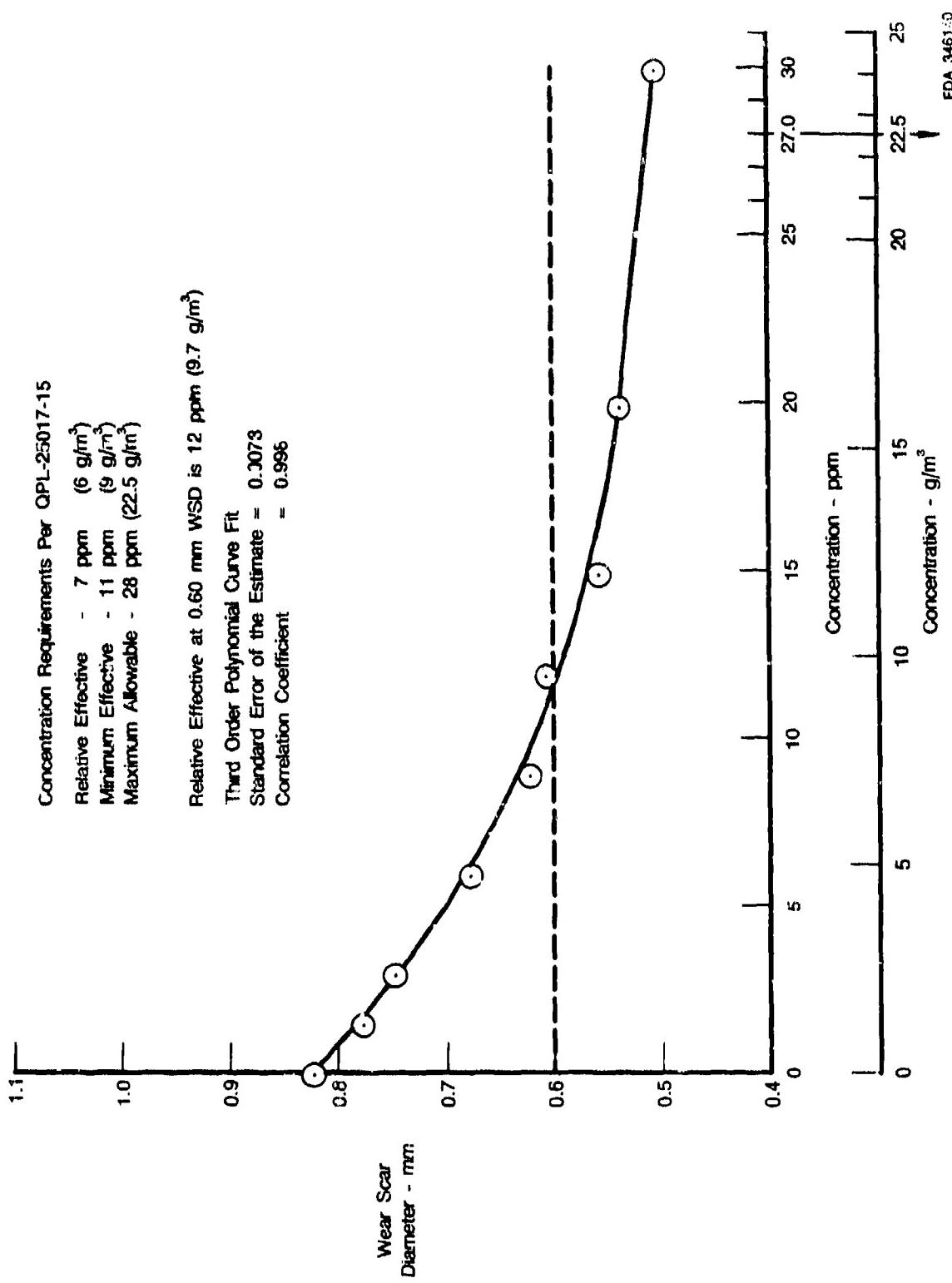


Figure D-37. — Effect of LUERIZOL 541 in Clay Treated JP-8



D-40

Figure D-38. — Effect of NALCO 5403 in Clay Treated JP-8

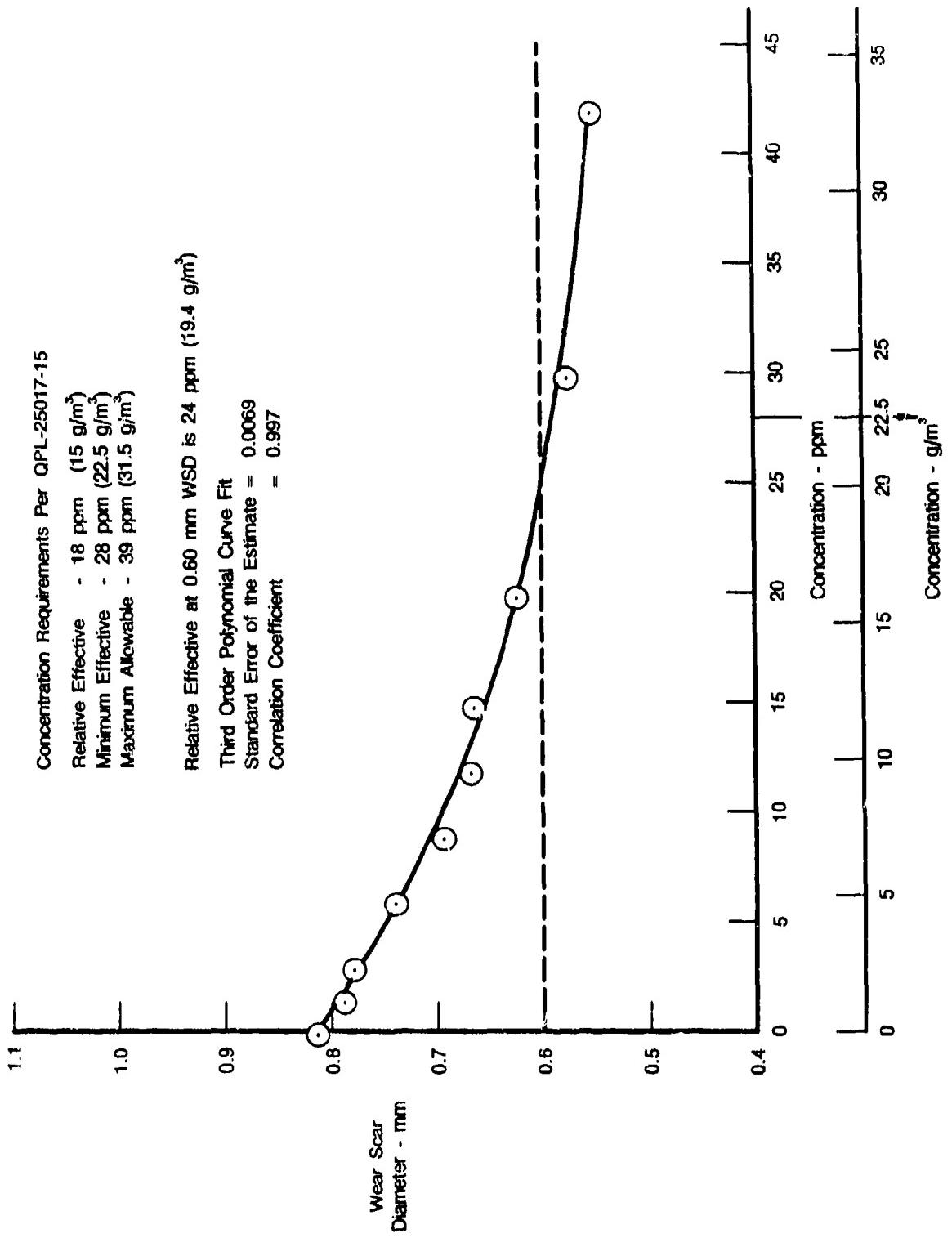


Figure D-39. — Effect of TOLAD 245 in Clay Treated JP-8

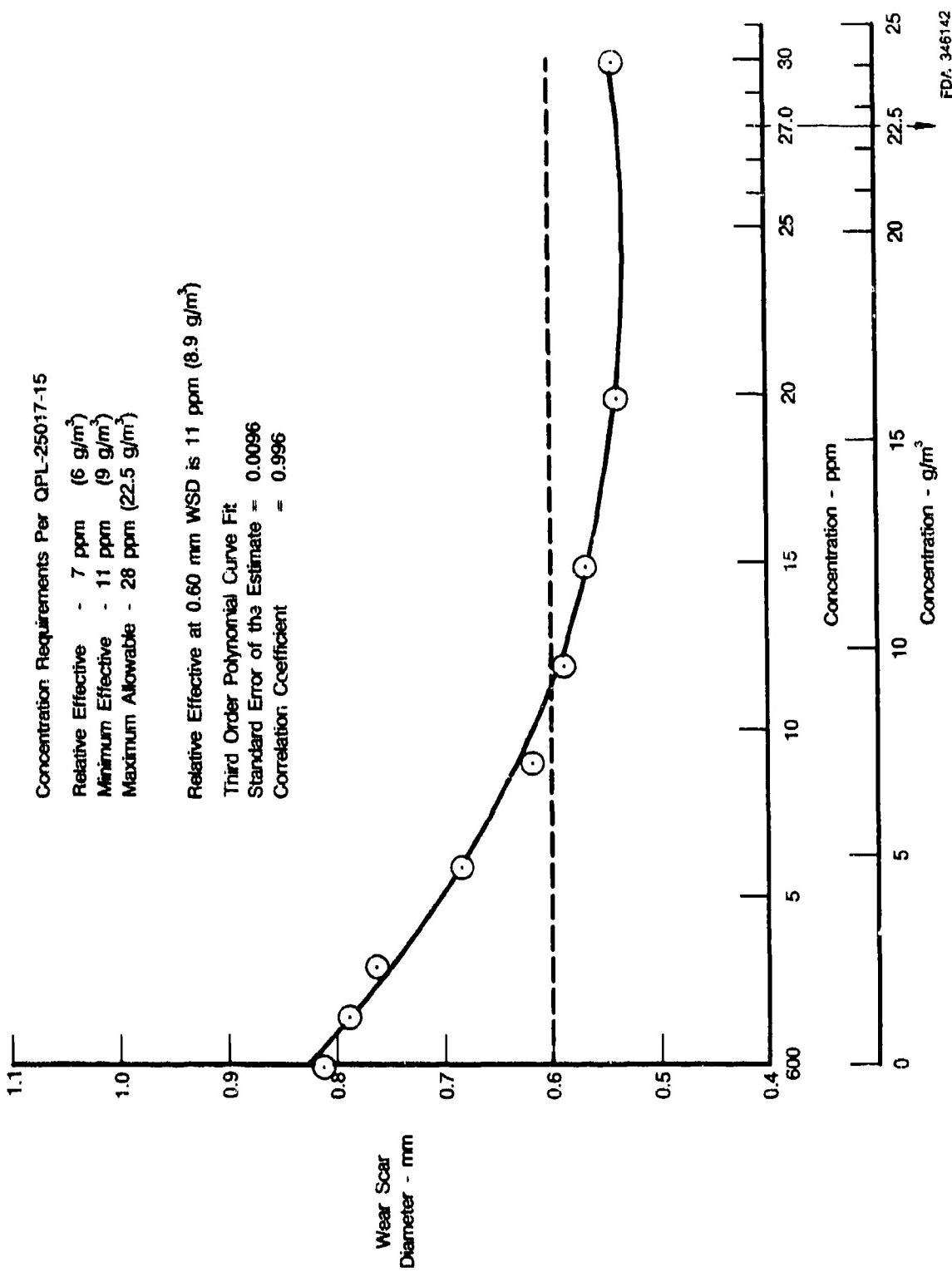


Figure D-40. — Effect of UNICOR-J in Clay Treated JP-8

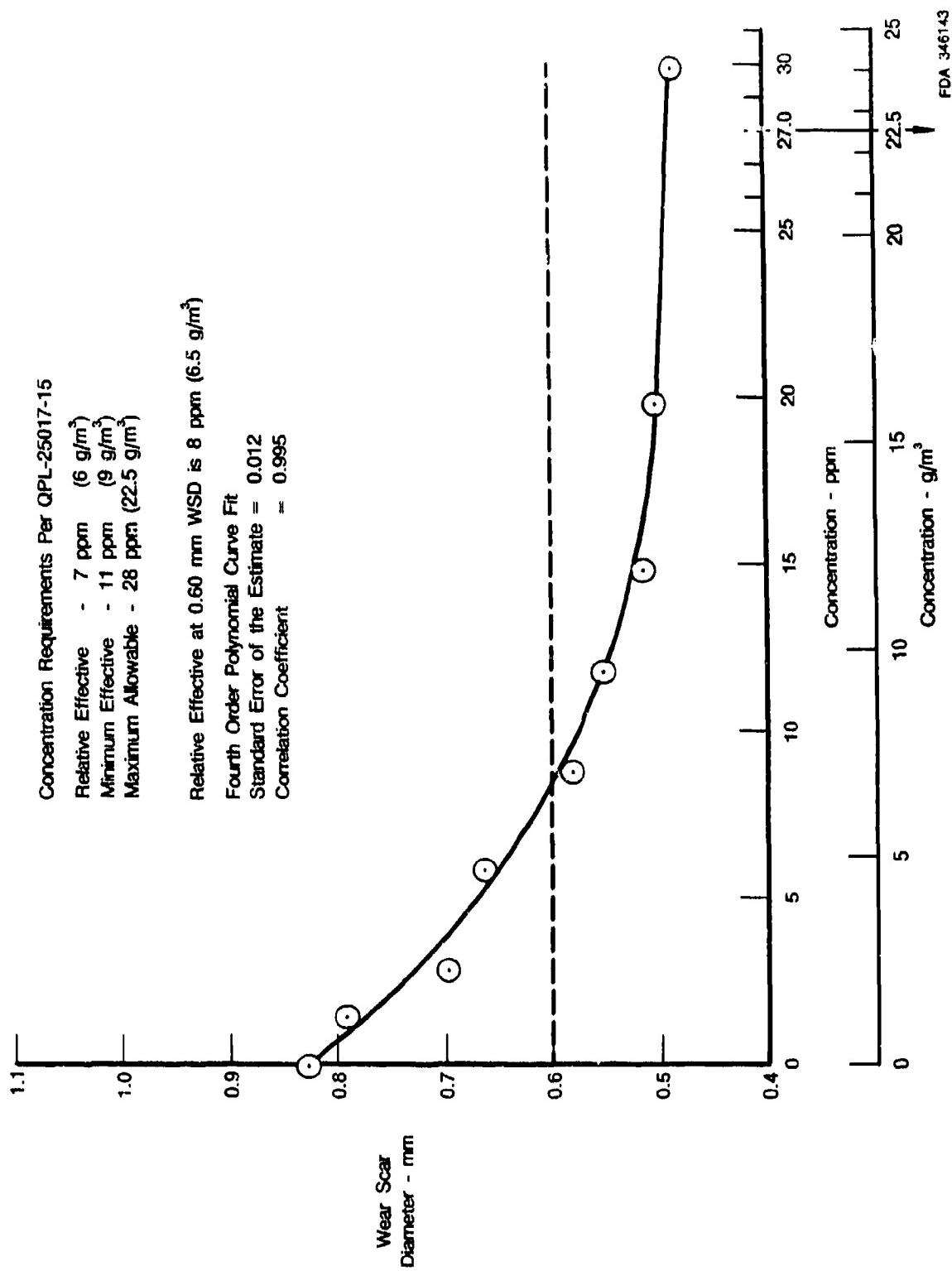


Figure D-41. — Effect of IPC 4410 in Clay Treated JP-8

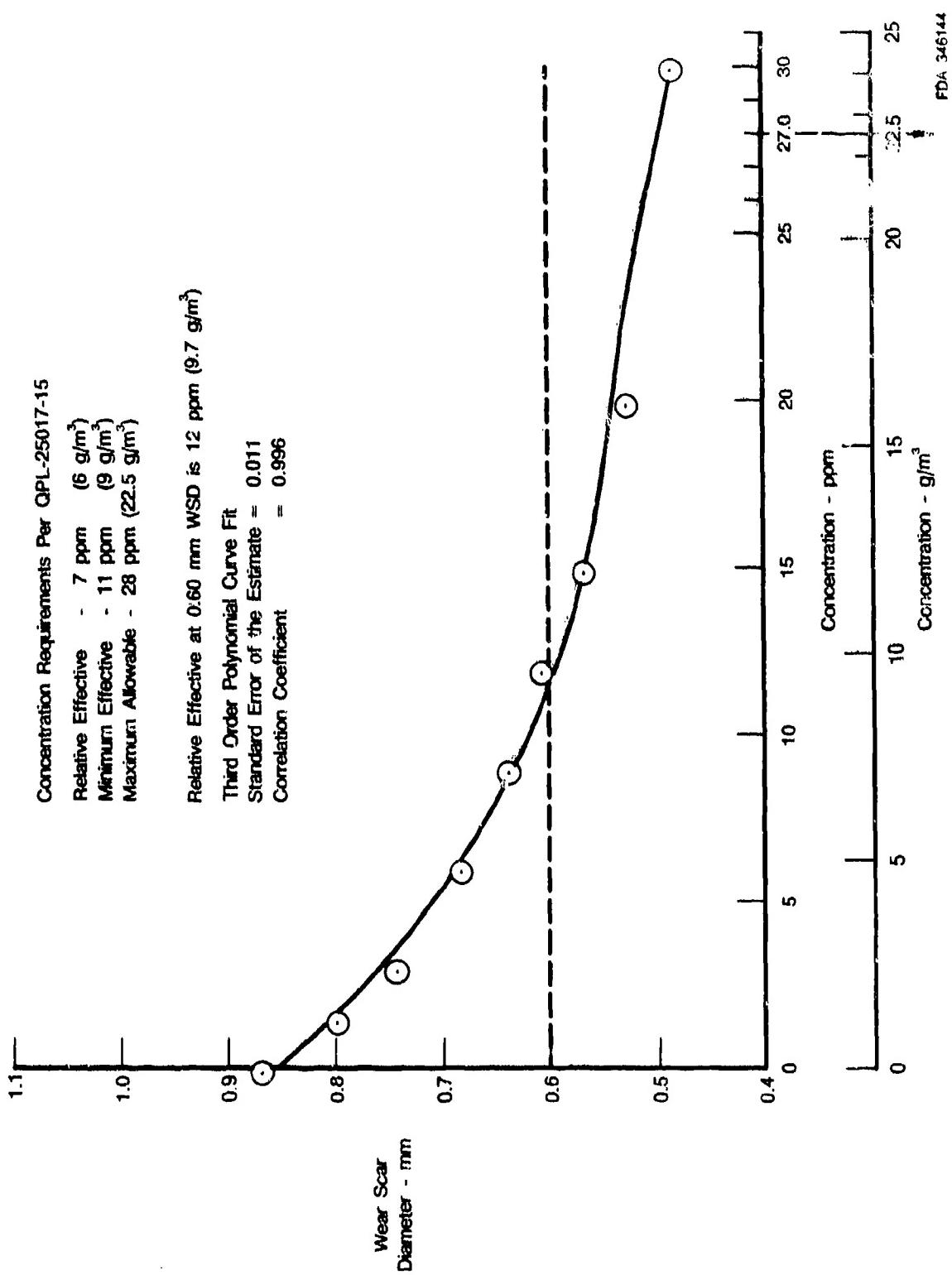


Figure D-42. — Effect of MOBILAD F-800 in Clay Treated JP-8

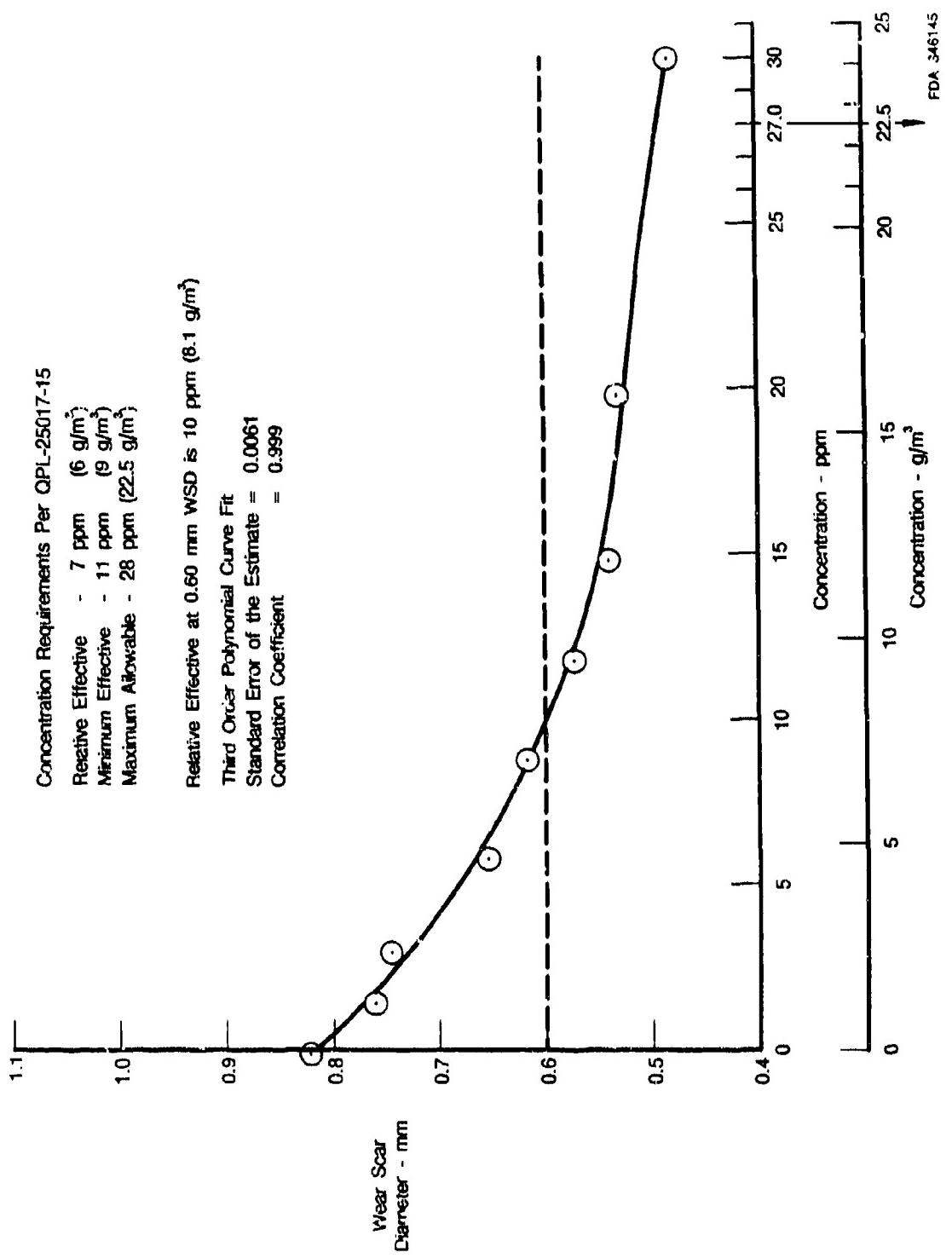


Figure L-43. — Effect of NALCO 5405 in Clay Treated JP-8

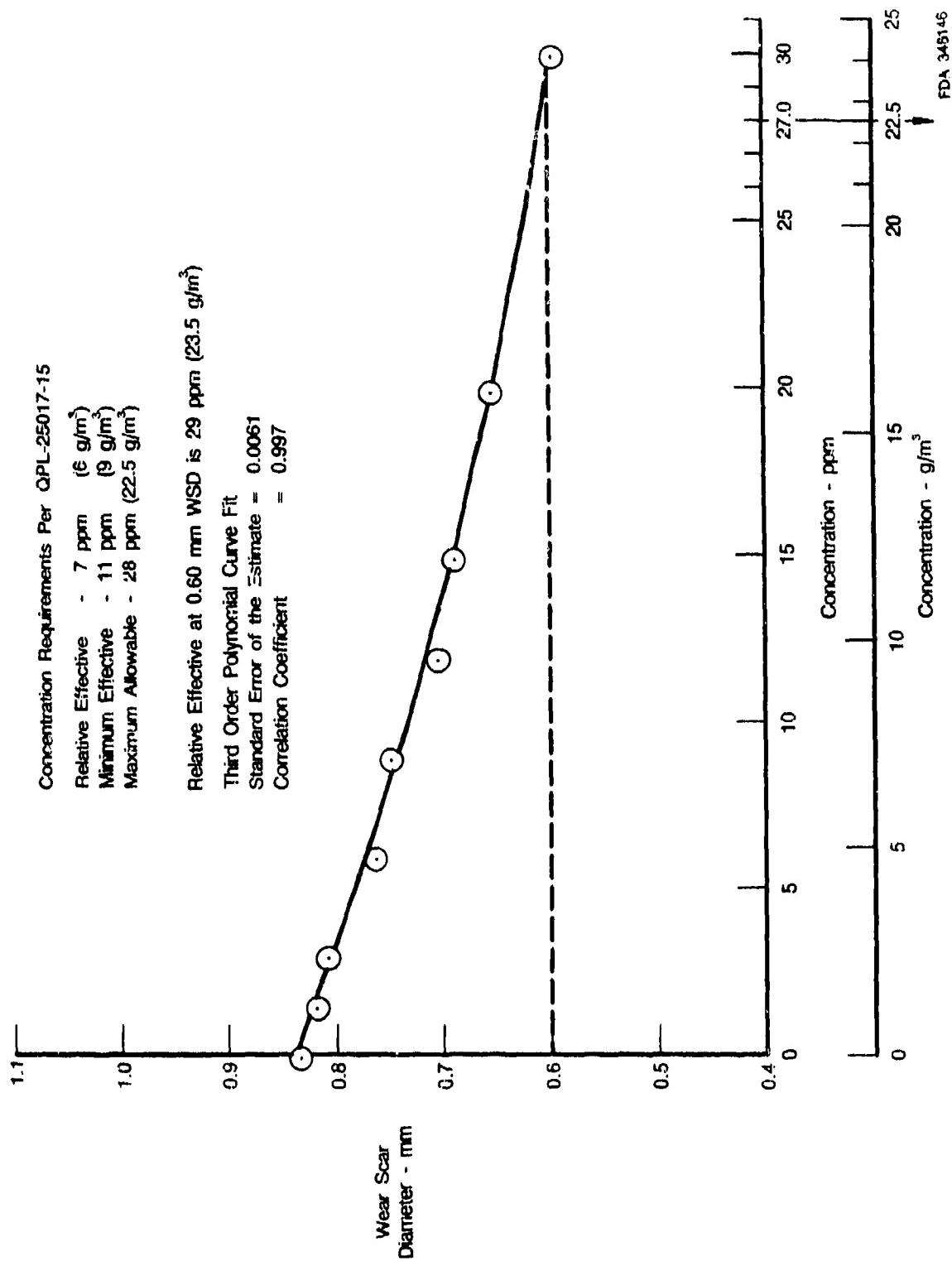


Figure D-44. — Effect of TOLAD 249 in Clay Treated JP-8

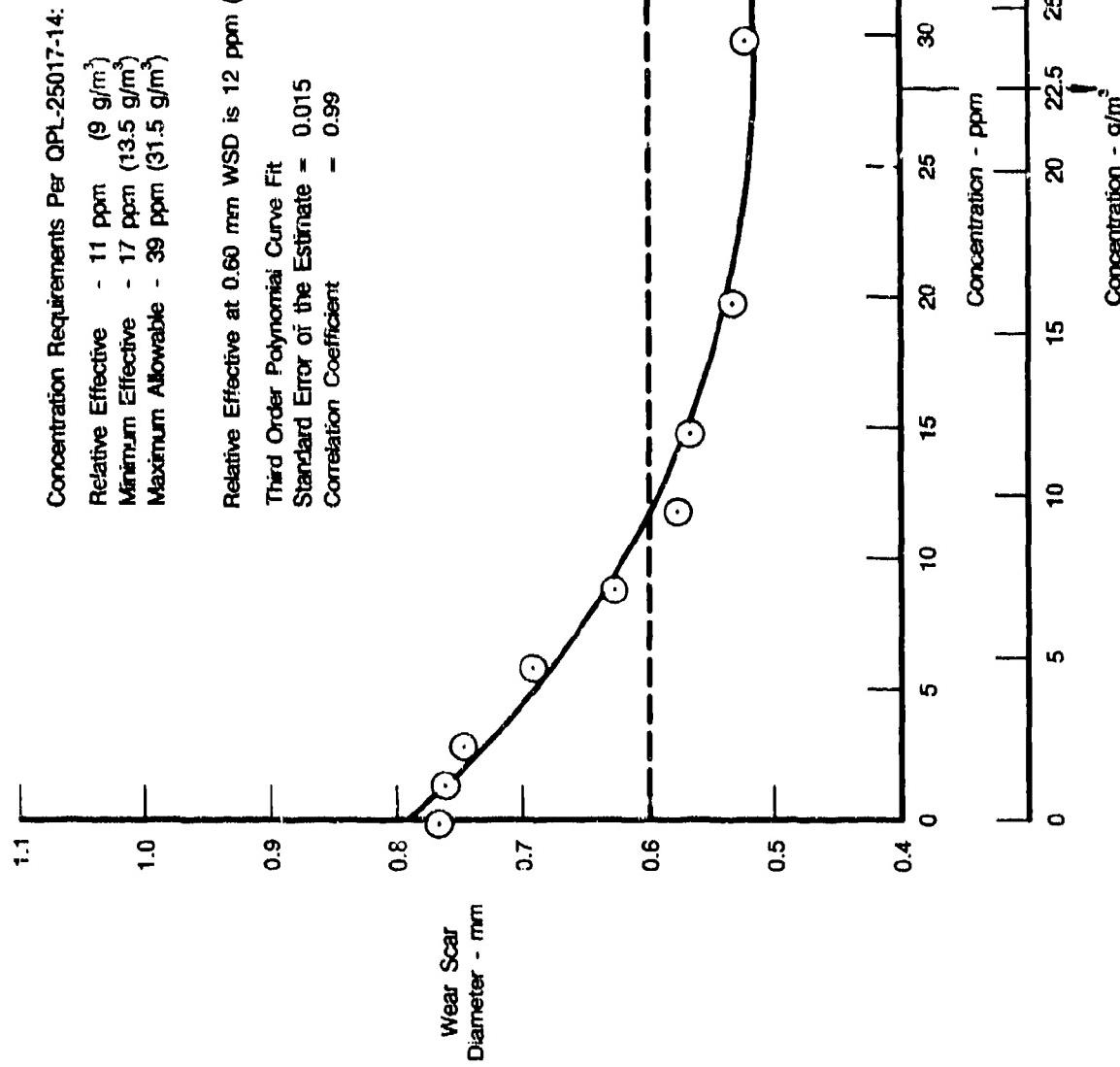


Figure D-45. — Effect of P-3305 in Clay Treated JP-8

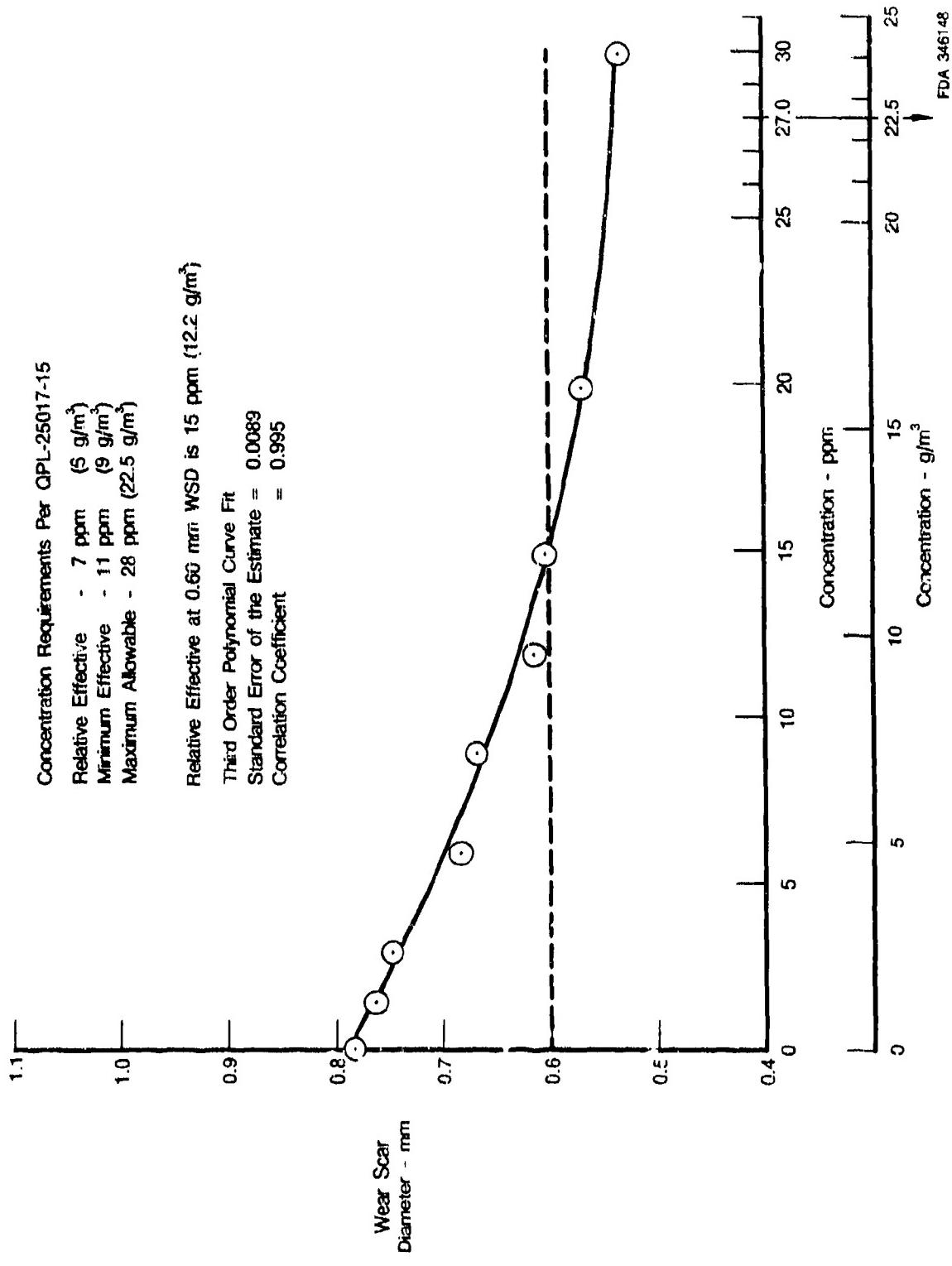


Figure D-46. — Effect of IPR-4445 in Clay Treated JP-8

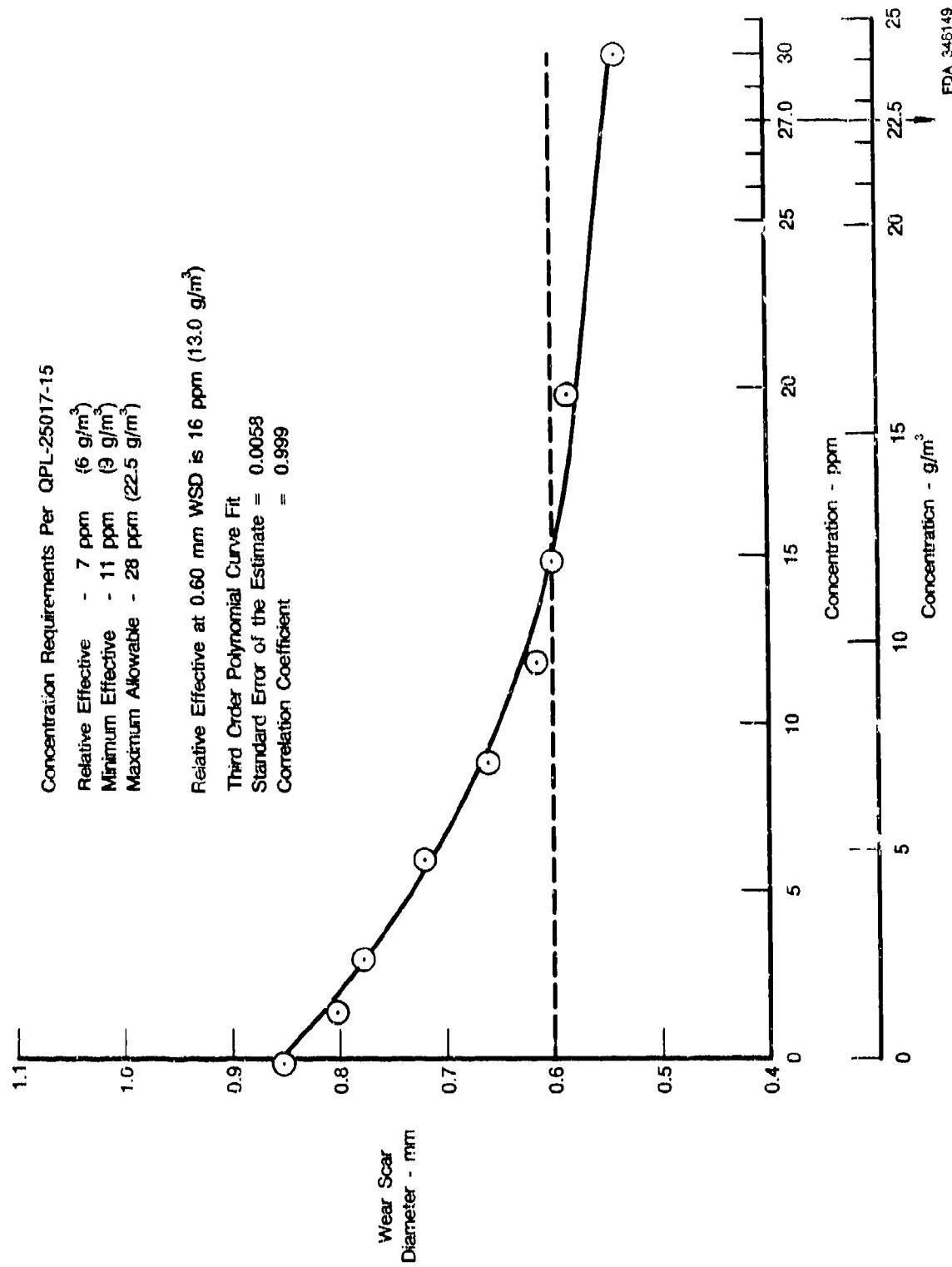


Figure D-47. — Effect of WELCHEM 91120 in Clay Treated JP-8

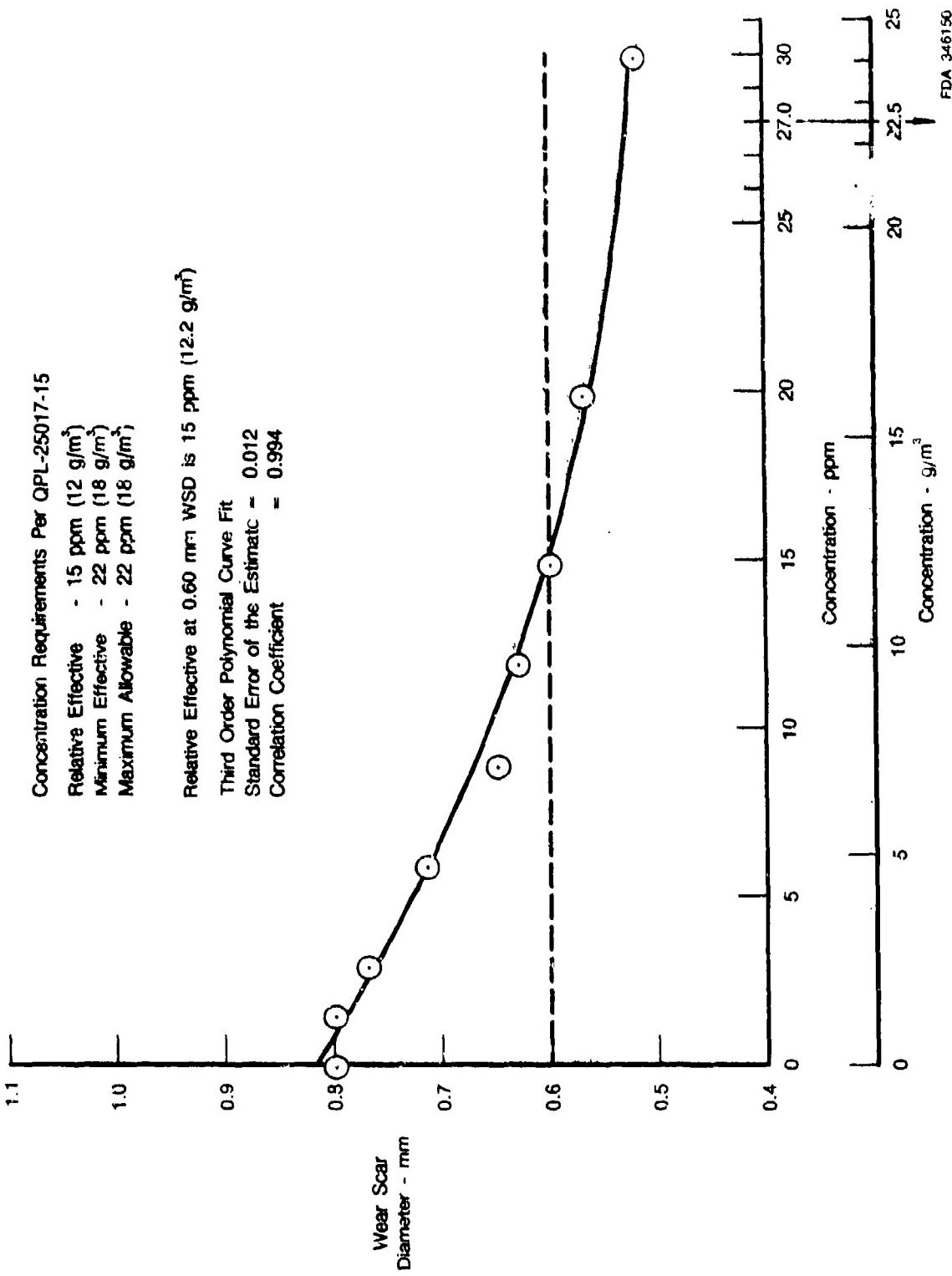


Figure D-48. — Effect of NUCHEM PCI-105 in Clay Treated JP-8

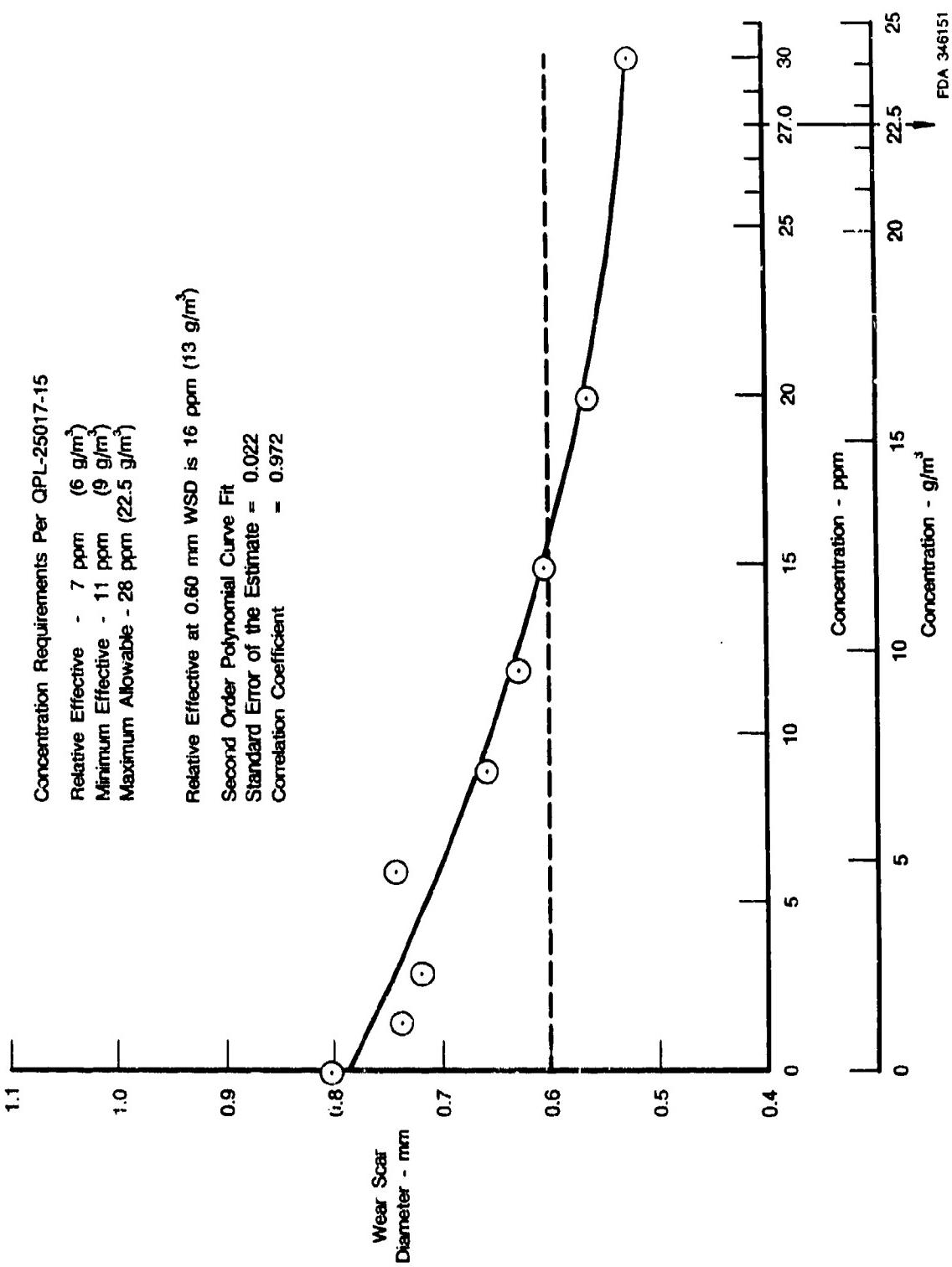


Figure D-49. — Effect of Apollo PRI-19 in Clay Treated JP-8

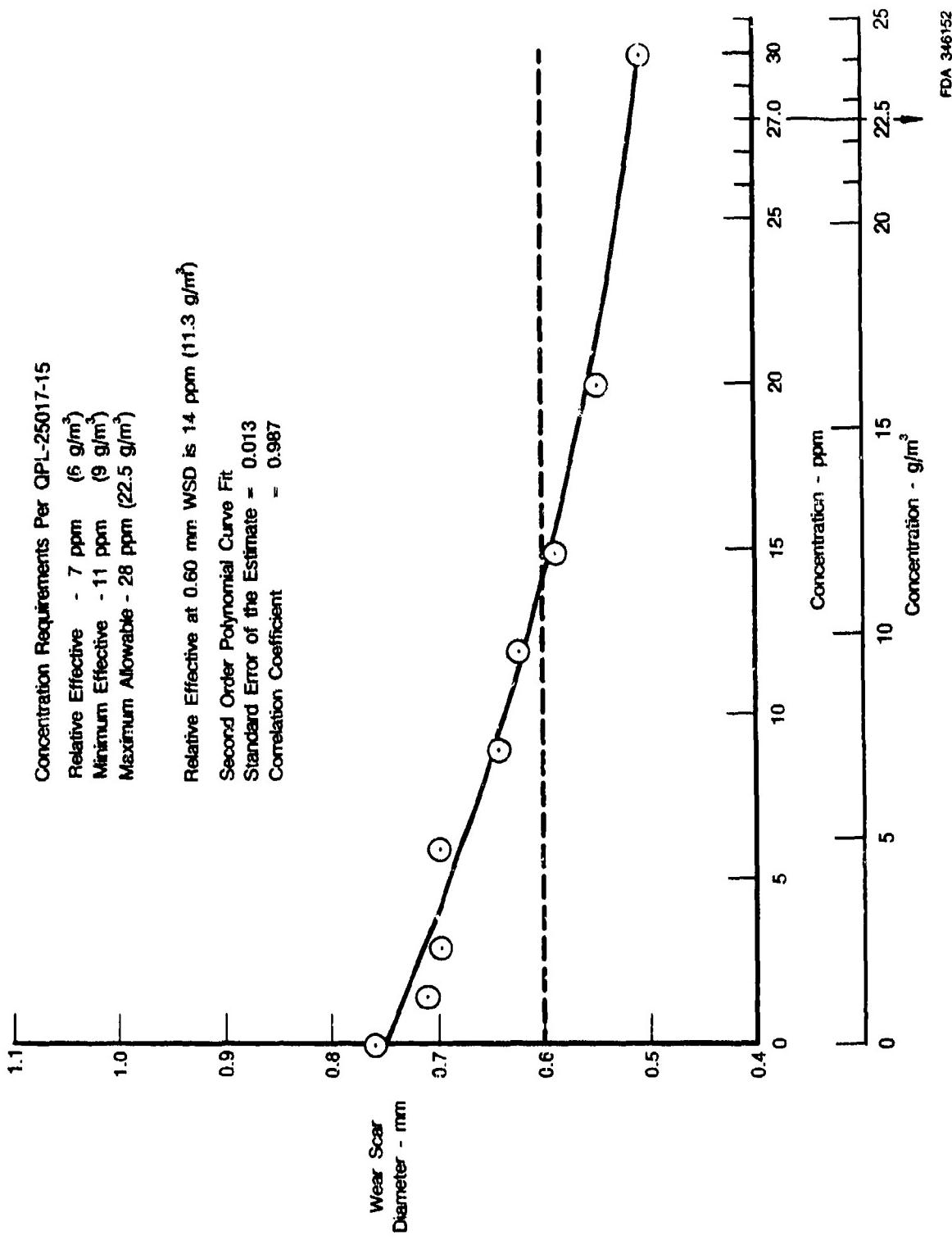


Figure D-50. — Effect of HITEC E-580 in Clay Treated JP-5

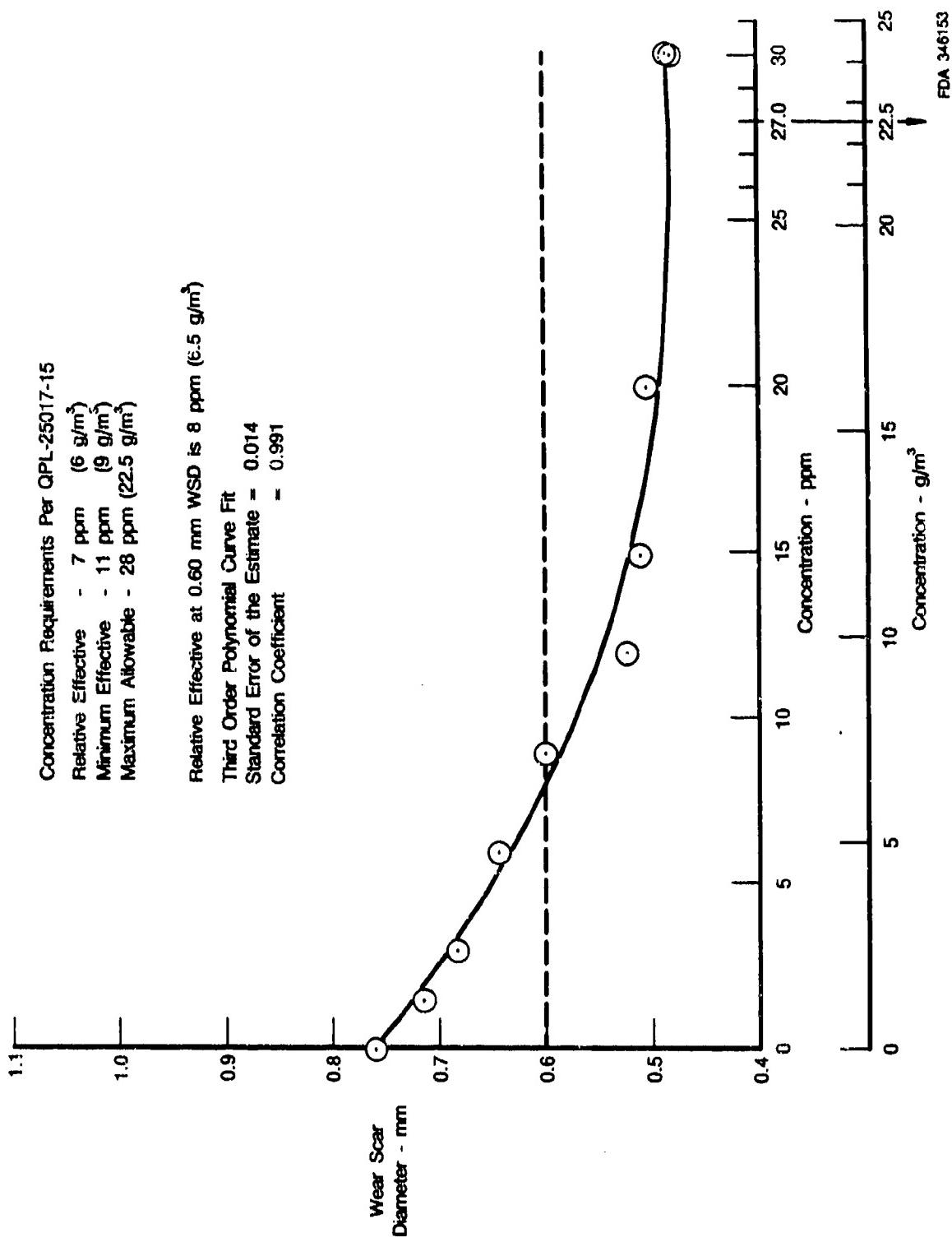


Figure D-51. - Effect of DCI-4A in Clay Treated JP-5

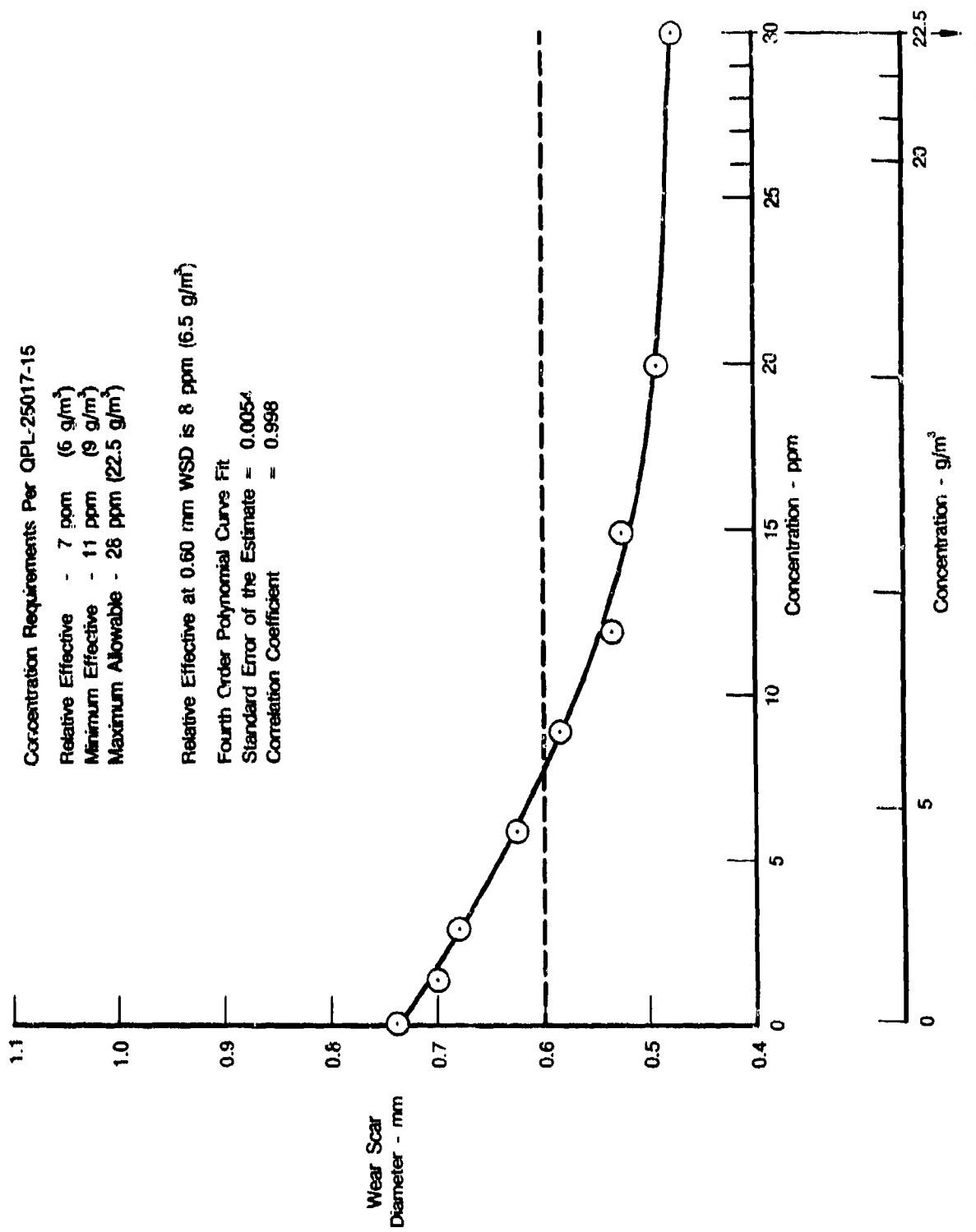


Figure D-52. — Effect of DCI-6A in Clay Treated JP-5

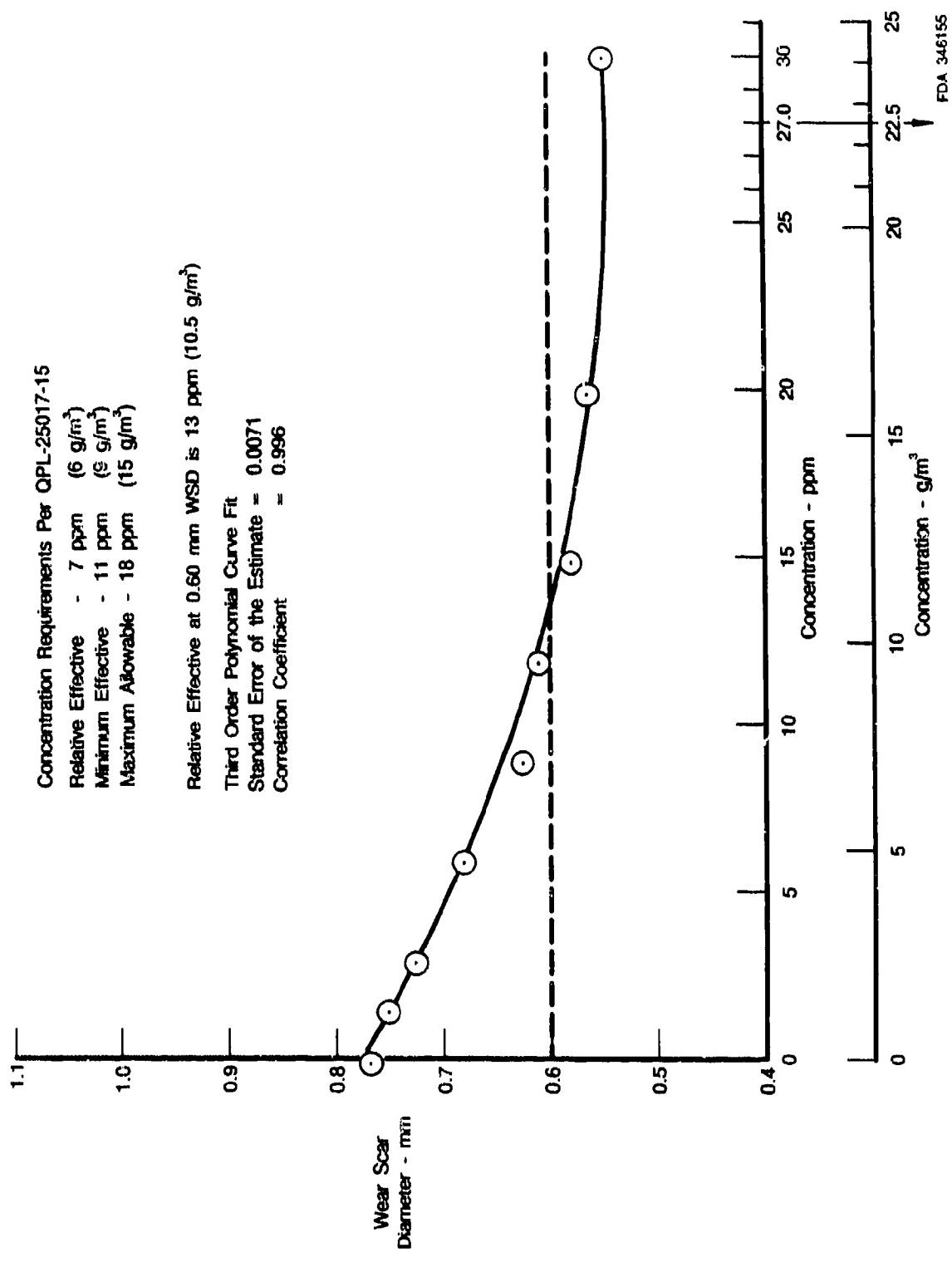


Figure D-53. — Effect of LUBRIZOL 54I in Clay Treated JP-5

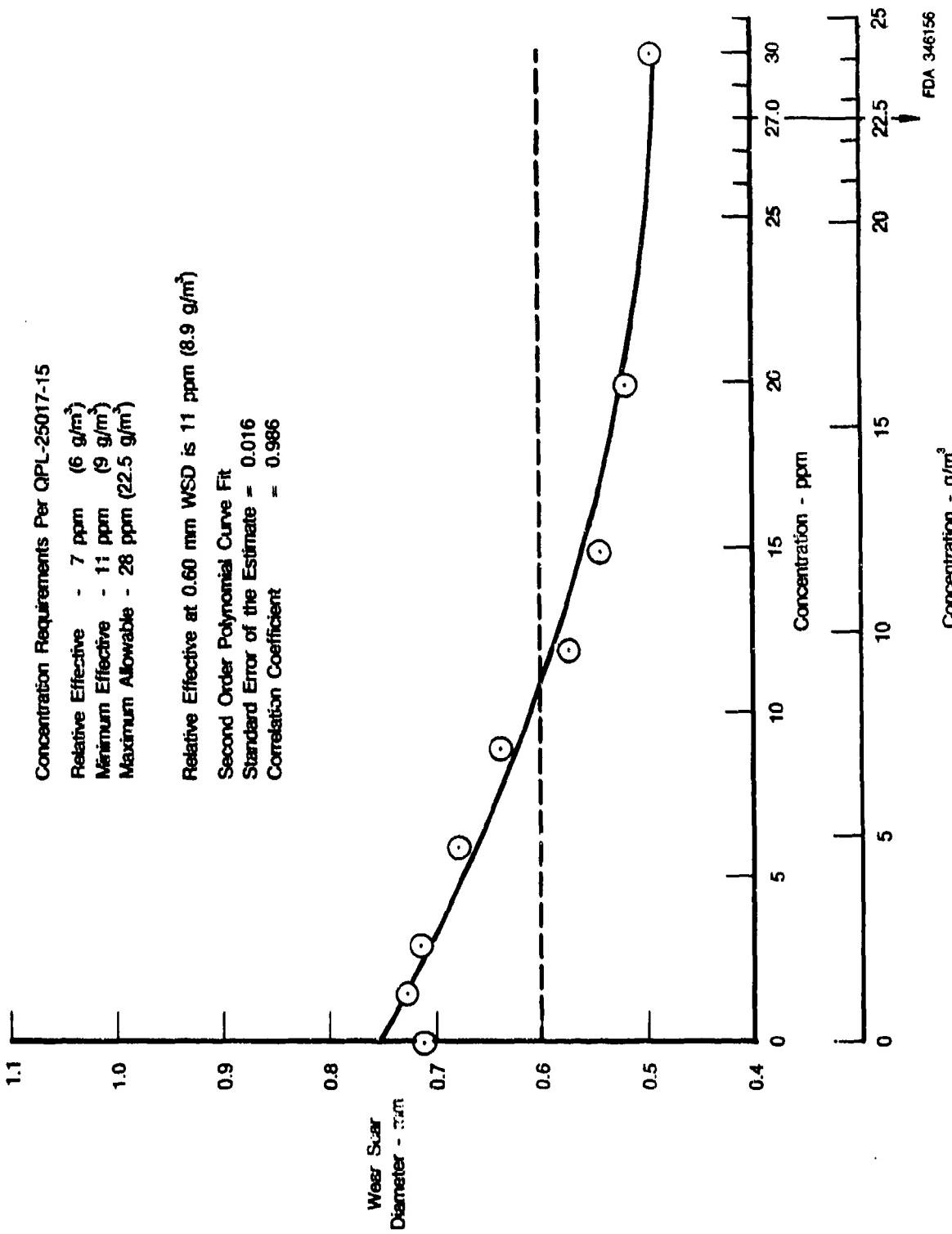


Figure D-54. — Effect of NALCO 5403 in Clay Treated JP-5

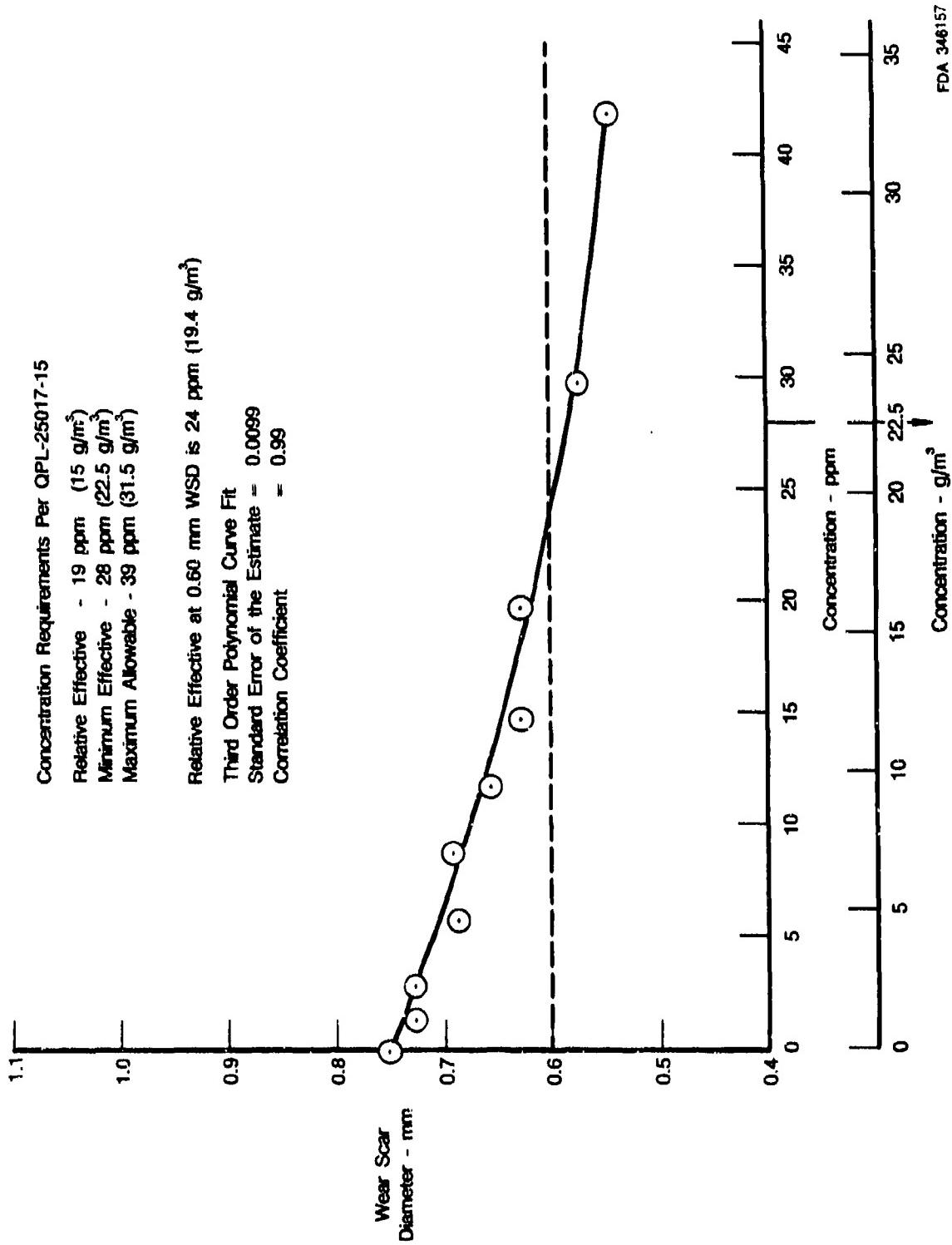


Figure D-55. — Effect of TOLAD 245 in Clay Treated JP-5

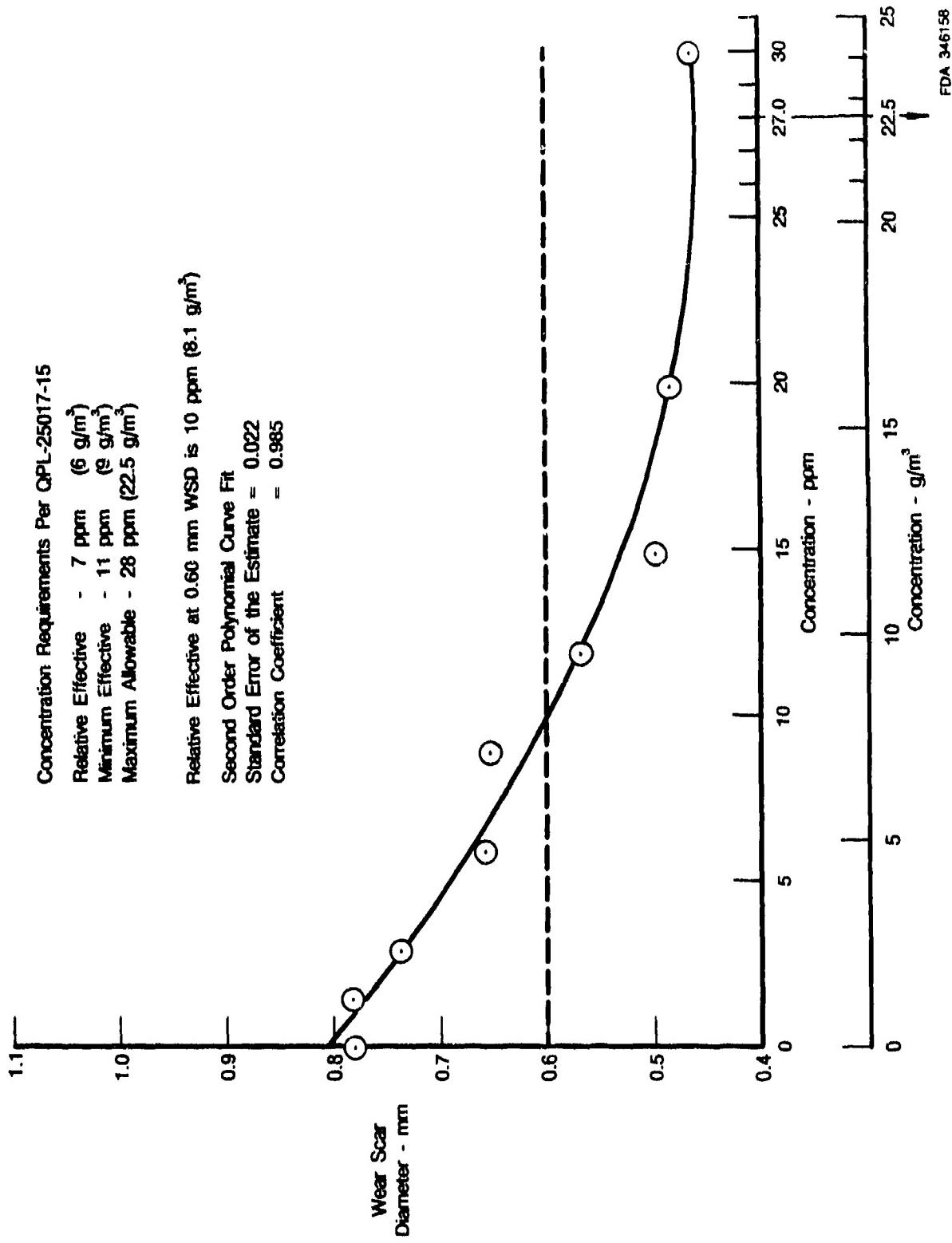


Figure D-56. — Effect of UNICOR-J in Clay Treated JP-5

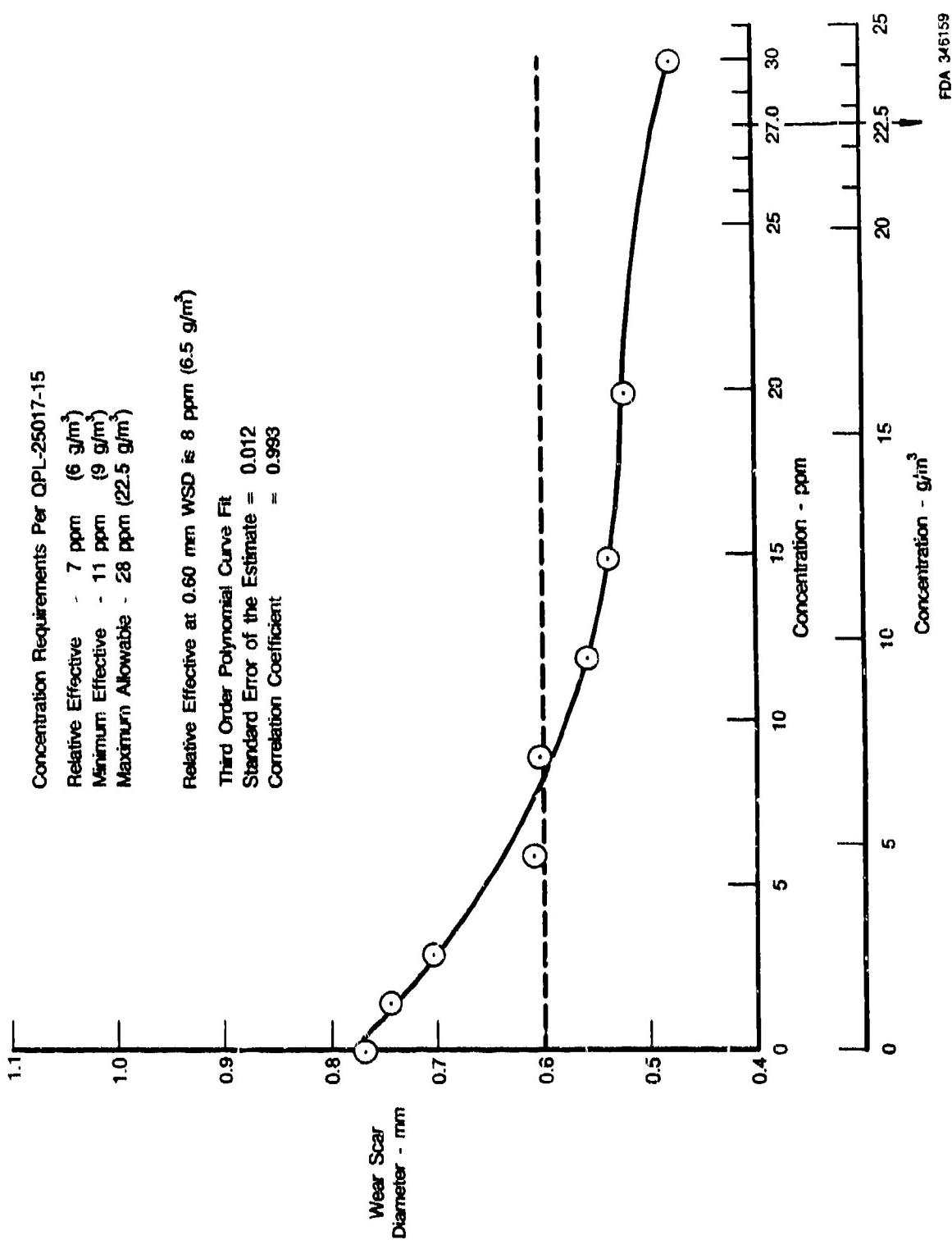


Figure D-57. — Effect of IIC-4410 in Clay Treated JP-5

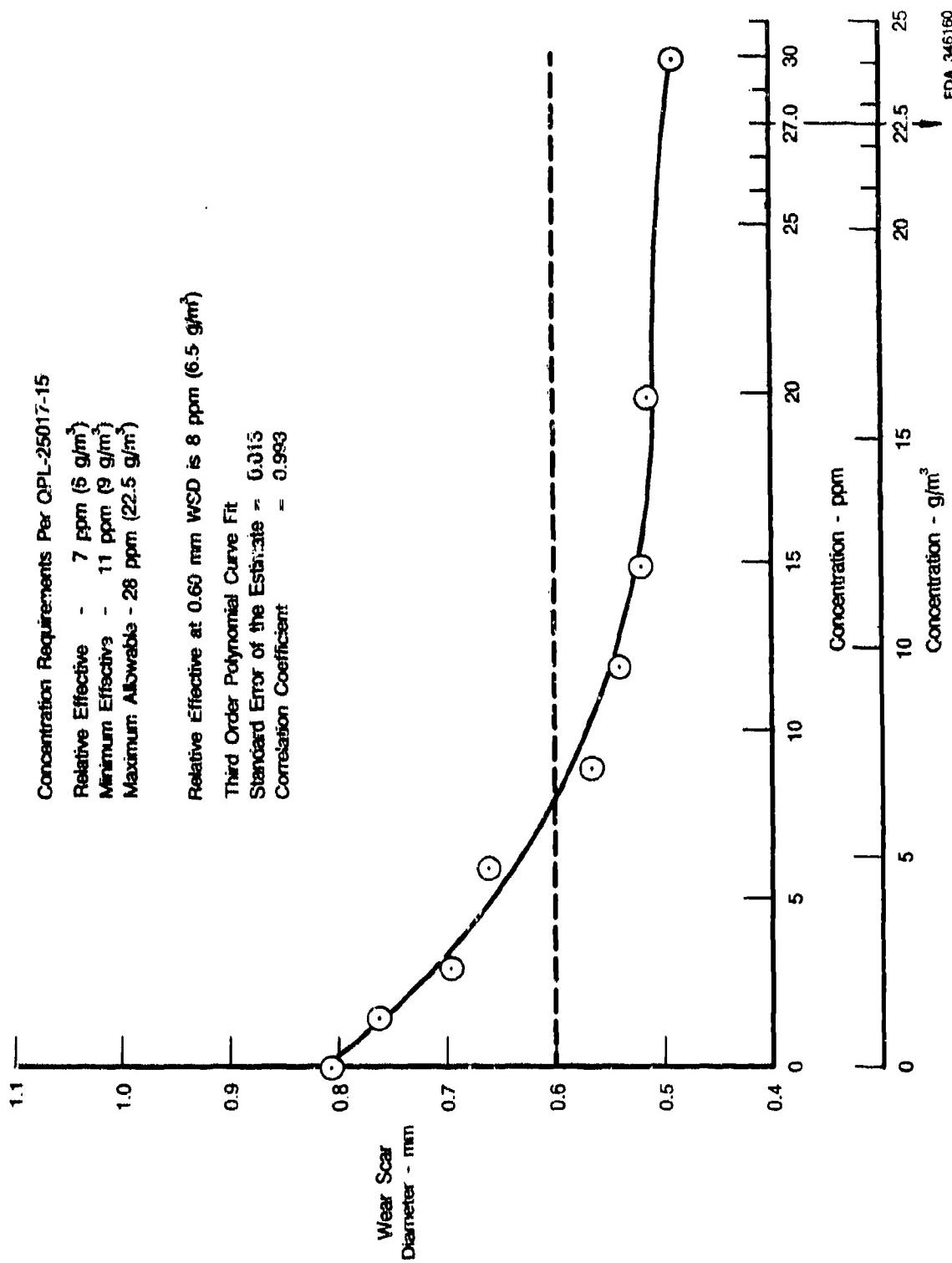


Figure D-58. — Effect of MOBILAD F-800 in Clay Treated JP-5

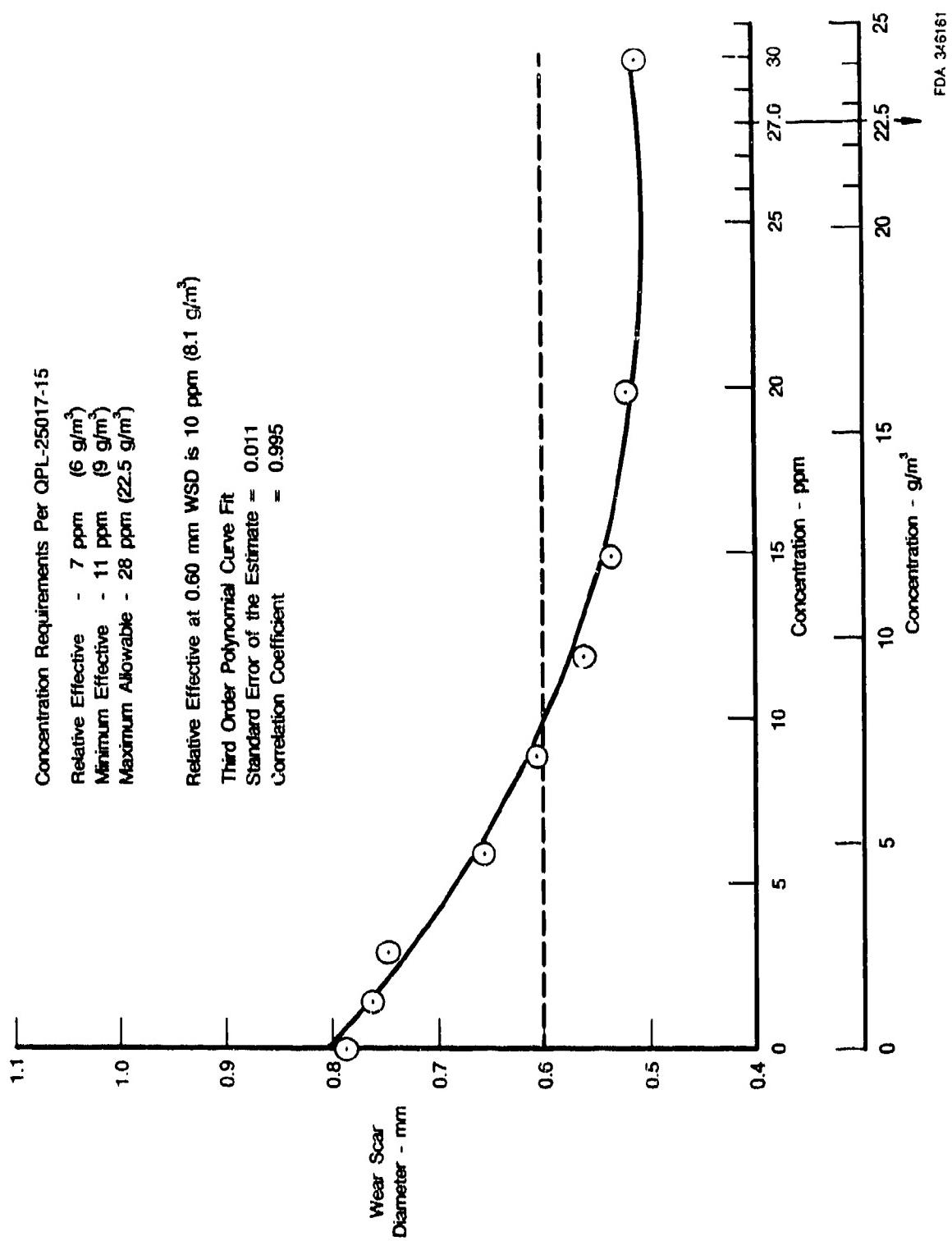


Figure D-59. — Effect of NALCO 5405 in Clay Treated JP-5

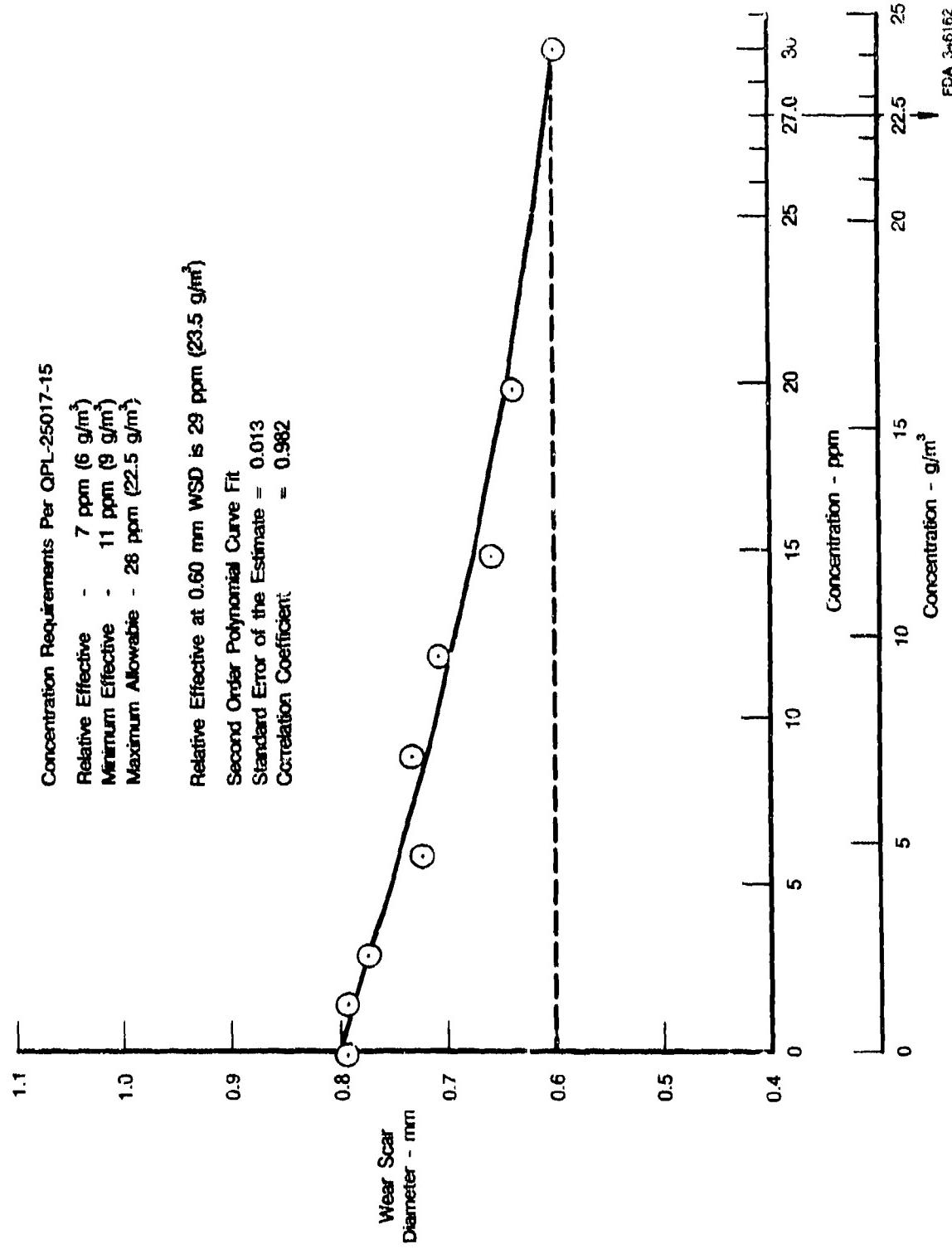


Figure D-60. — Effect of TOLAD 249 in Clay Treated JP-5

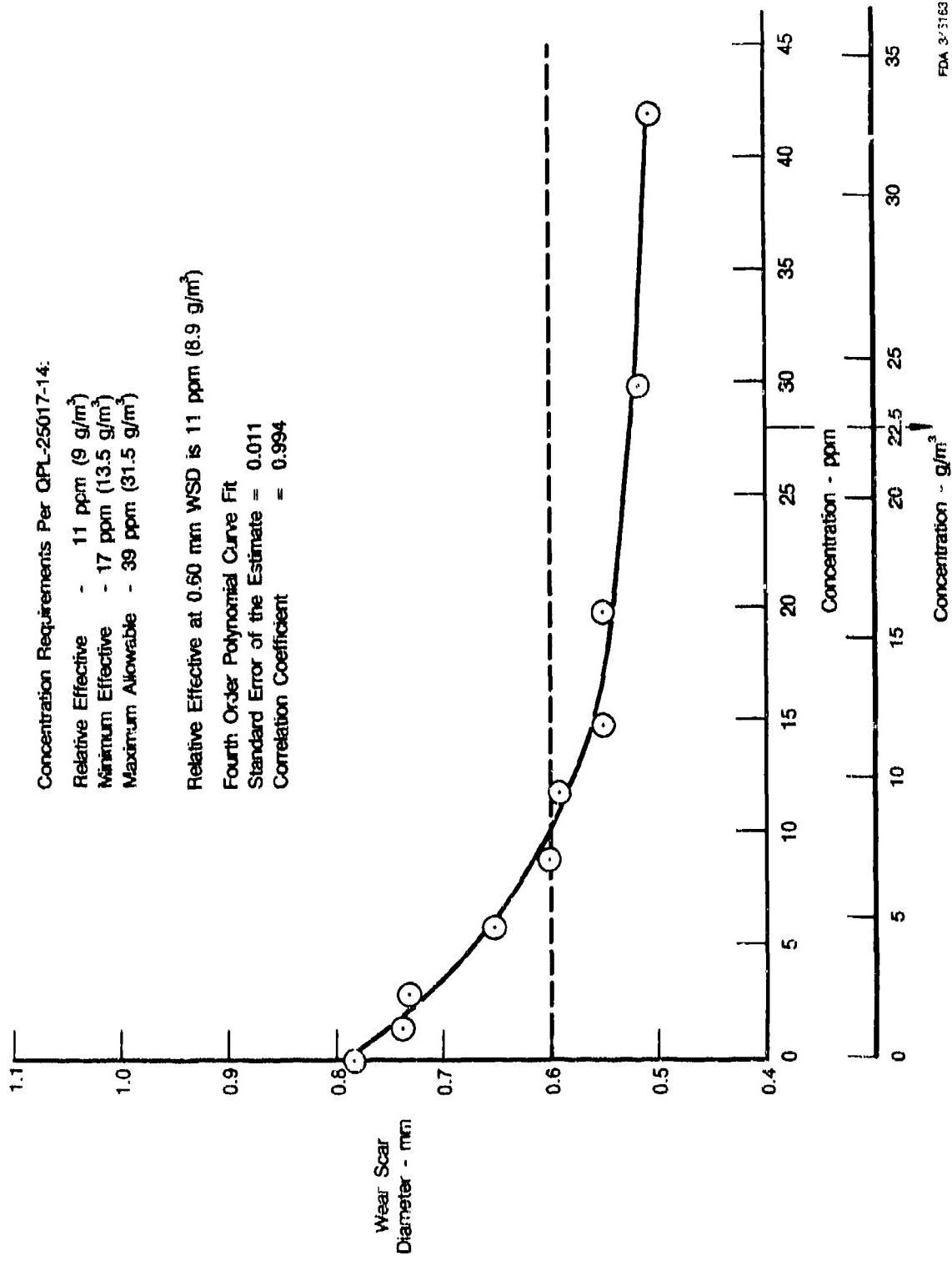
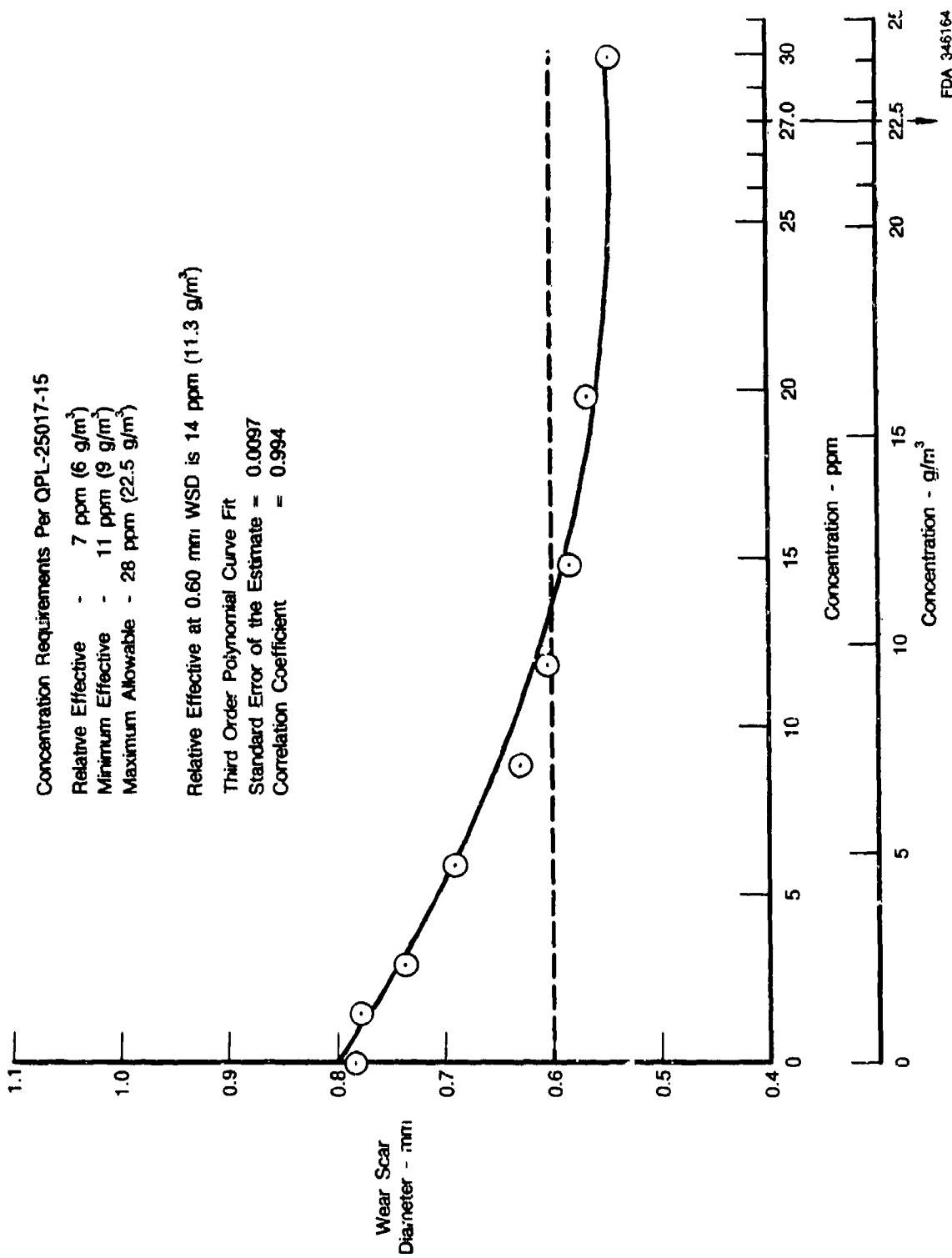


Figure D-61. — Effect of P-3395 in Clay Treated JP-5



D-64

Figure D-62. — Effect of IPC-445 in Clay Treated JP-5

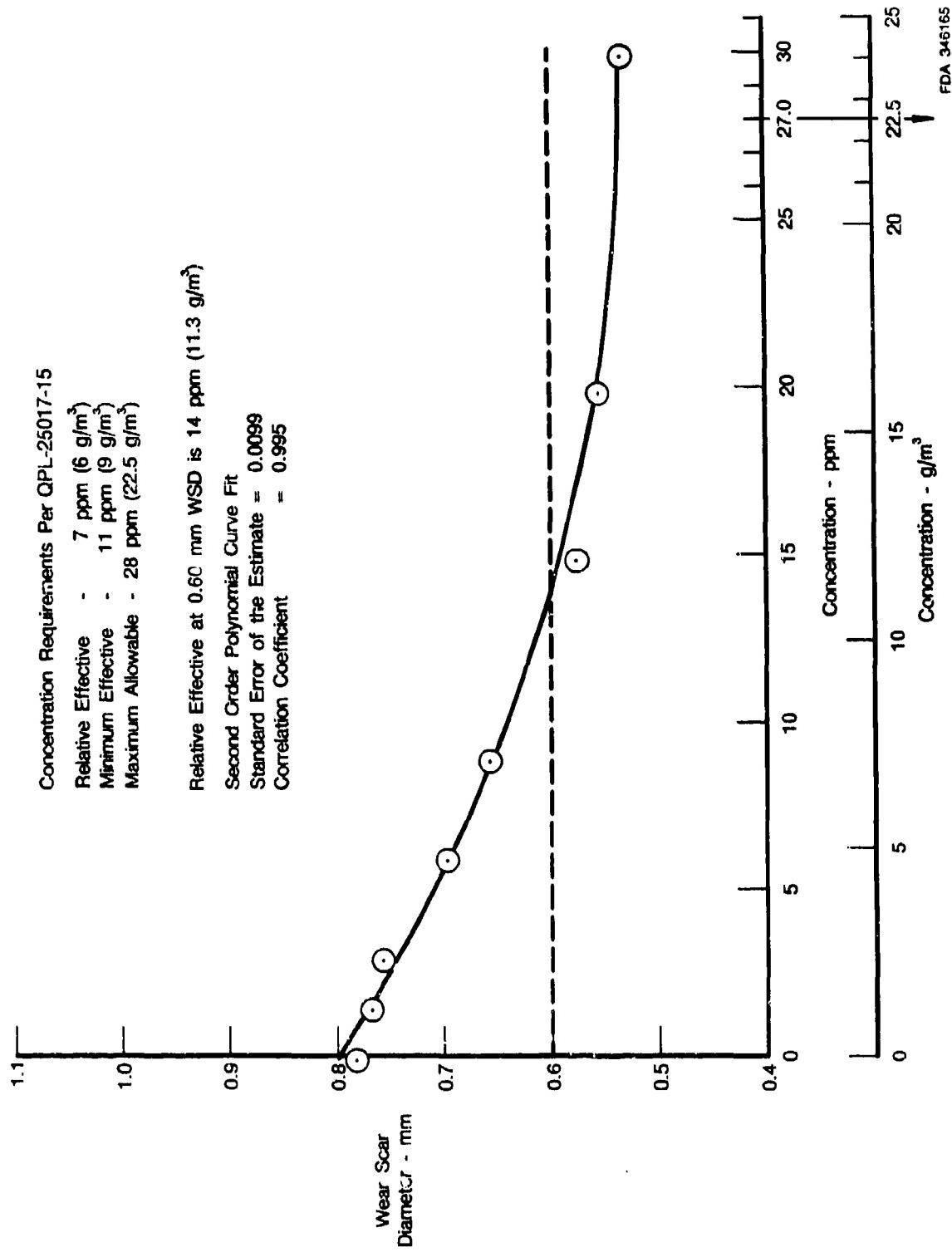


Figure D-63. — Effect of WELCHEM 91120 in Clay Treated JP-5

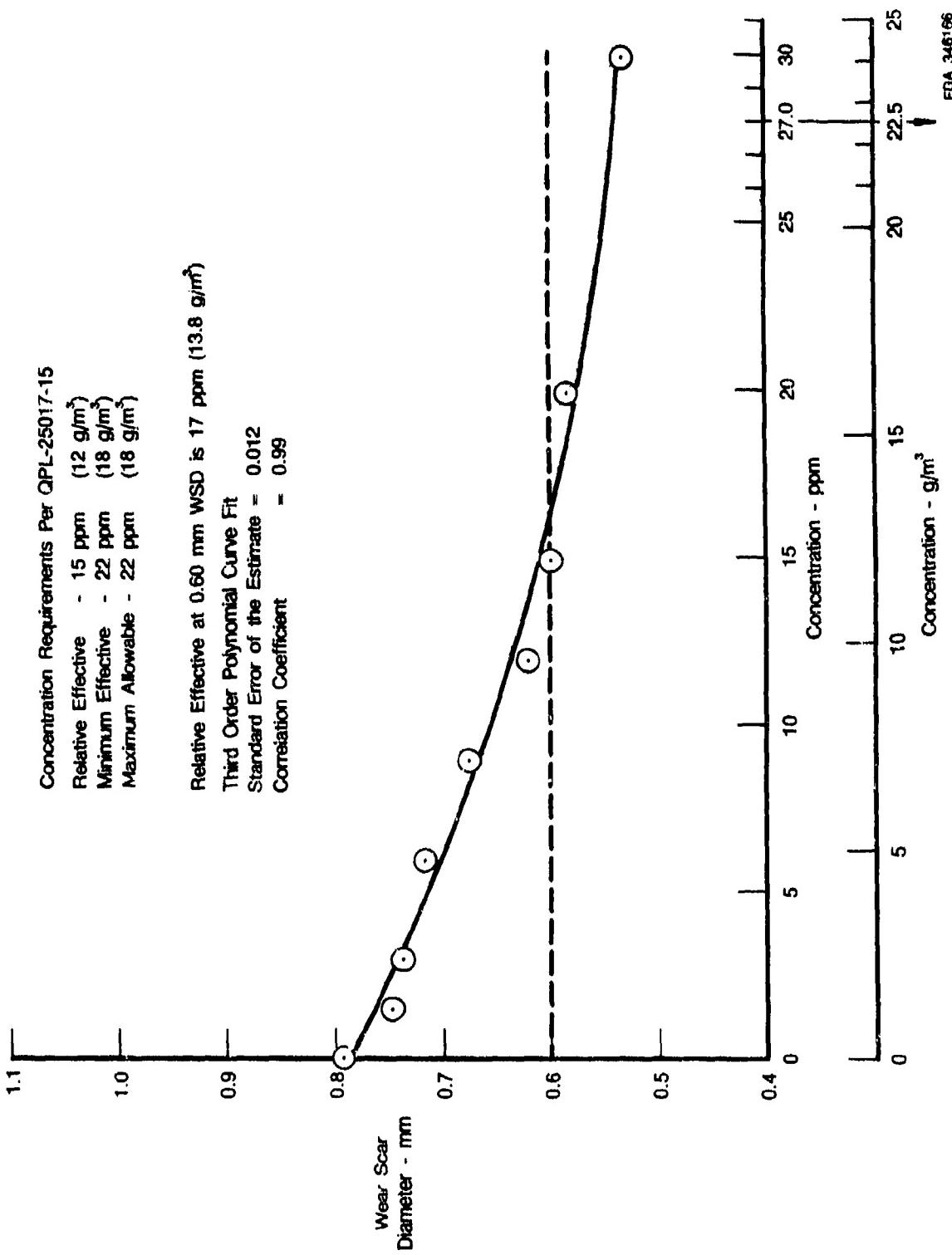


Figure D-64. — Effect of NUCHEM PCI-105 in Clay Treated JP-5

MIL-I-25017D
27 May 1981
Superseding
MIL-I-25017C
8 March 1971

APPENDIX E

INHIBITOR, CORROSION/LUBRICITY IMPROVER, FUEL SOLUBLE (METRIC)

This specification is approved for use by all
Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers one type of fuel soluble corrosion inhibitor/lubricity improver additive for use in aviation turbine fuel, motor gasoline, diesel fuel, and related petroleum products.

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

Federal

TT-S-735	Standard Test Fluids; Hydrocarbon
VV-F-0800	Fuel Oil, Diesel
VV-G-1690	Gasoline, Automotive, Leaded or Unleaded

Military

MIL-G-3056	Gasoline, Automotive, Combat
MIL-T-5624	Turbine, Fuel, Aviation Grades JP-4 and JP-5
MIL-C-7024	Calibrating Fluid, Aircraft Fuel System Components
MIL-L-7808	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base
MIL-F-25558	Fuel, Ramjet Engine, Grade RJ-1
MIL-I-27686	Inhibitor, Icing, Fuel System
MIL-G-46015	Gasoline, Automotive, Combat, Referee Grade
MIL-F-46162	Fuel, Diesel, Referee Grade
MIL-T-83133	Turbine Fuel, Aviation, Kerosene Type, Grade JP-8

STANDARDS

Federal

FED-STD-791 Lubricants, Liquid Fuels, and Related Products;
Methods of Testing

Military

MIL-STD-290 Packaging of Petroleum and Related Products

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents from a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials

ASTM A 108	Cold-Finished Carbon Steel Bars and Shafting
ASTM D 56	Test for Flash Point by Tag Closed Tester
ASTM D 97	Test for Pour Point of Petroleum Oils
ASTM D 270	Sampling Petroleum and Petroleum Products
ASTM D 445	Test for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
ASTM D 482	Test for Ash from Petroleum Products
ASTM D 664	Test for Neutralization Number by Potentiometric Titration
ASTM D 665	Test for Rust-Preventing Characteristics of Steam- Turbine Oil in the Presence of Water

ASTM D 1298	Test for Density, Specific Gravity, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
ASTM D 2274	Test for Stability of Distillate Fuel Oil (Accelerated Method)
ASTM D 2550	Test for Water Separation Characteristics of Aviation Turbine Fuels
ASTM D 2624	Test for Electrical Conductivity of Aviation Turbine Fuels Containing a Static Dissipator Additive
ASTM D 3114	Test for D-C Electrical Conductivity of Hydrocarbon Fuels

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19013.)

Manufacturing Chemist's Association Incorporated

Manual L-1	Warning Labels — A Guide for the Preparation of Warning Labels for Hazardous Chemicals
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(Application for copies should be addressed to the Manufacturing Chemists' Association, Incorporated, 1835 Connecticut Avenue, N.W., Washington, DC 20009.)

3. REQUIREMENTS

3.1 Qualification. The inhibitors furnished under this specification are for use in aviation turbine fuels, motor gasoline, diesel fuel, and related petroleum products. The inhibitor shall be a product that has passed the applicable qualification tests listed below and has been listed on or approved for listing on the applicable qualified products list. Tentative approval for listing on the qualified products list shall be granted pending successful completion of the storage stability tests. Failure to pass the storage stability requirement of 3.12 shall be cause for withdrawal of approval.

3.1.1 Qualification requirements. The qualification requirements for the inhibitors are listed for each type of fuel. All approved inhibitors shall meet the requirements of 3.2 through 3.12, 3.16, and 3.17 to be qualified for use in fuels conforming to MIL-T-5624, MIL-C-7024, MIL-F-25558, and MIL-T-83133. To qualify for use in motor gasolines (MIL-G-3056 and VV-G-1690) and diesel fuel (VV-F-800), the inhibitors shall also pass the applicable requirements of 3.13 through 3.15.

3.2 Materials. The composition of the finished inhibitor is not limited but is subject to review by the qualifying activity in order to assure service compatibility with previously qualified products.

3.2.1 Toxic products and formulations. The material shall have no adverse effect on the health of personnel when used for its intended purpose. Questions pertinent to this effect shall be referred by the procuring activity to the appropriate departmental medical service who will act as an advisor to the procuring activity.

3.3 Solubility. The maximum allowable concentration of inhibitor, as defined in 3.7, shall be readily and completely dissolved in all fuels for which it is qualified. There shall be no precipitation, cloudiness or other evidence of insolubility when tested as specified in 4.6.1.

3.4 Compatibility. The inhibitor shall be compatible with all inhibitors currently qualified under this specification and with the static dissipator additives listed in MIL-T-5624. There shall be no precipitation, cloudiness, other evidence of noncompatibility when tested as specified in 4.6.2.

3.5 Relative effective concentration. The relative effective concentration shall be determined in accordance with 4.6.3 and shall be expressed in grams of finished inhibitor per cubic meter of fuel. The relative effective concentration shall not be less than 6 grams of inhibitor per cubic meter of fuel (g/m^3) and shall be approved at concentrations divisible by 3 (e.g., 6, 9, 12, 15, . . . 33, and $36 \text{ g}/\text{m}^3$).

3.6 Minimum effective concentration. The minimum effective concentration shall be 1.5 times the relative effective concentration. This amount shall be not less than 9 grams of inhibitor per cubic meter of fuel.

3.7 Maximum allowable concentration. The maximum allowable concentration shall be the lowest of the following (all expressed in grams of inhibitor per cubic meter of fuel):

- a. Fifty-four grams of inhibitor per cubic meter of fuel
- b. Four times the relative effective concentration
- c. The highest concentration giving a Water Separometer Index Modified value of 70 or higher when determined in accordance with 4.6.4
- d. The highest concentration giving less than a 40% change in electrical conductivity with fuel containing static dissipator additive (see 4.6.2.2).

The maximum allowable concentration shall be equal to or greater than the minimum effective concentration and shall be a value evenly divisible by 4.5 within the range of 9 to $54 \text{ g}/\text{m}^3$.

3.8 Ash content. The ash content of the inhibitor shall not exceed 0.10 percent when determined in accordance with 4.6.5.

3.9 Pour point. The maximum allowable pour point of the finished inhibitor shall be -18°C when determined as specified in 4.6.6.

3.10 Aircraft turbine engine operation. Grade JP-4 fuel (MIL-T-5624) containing twice the maximum allowable concentration (see 3.7) of the inhibitor shall be tested in accordance with 4.6.7 to determine its acceptability for turbine engine use. Engine operation shall not be adversely affected and the post-test condition of the engine shall indicate no excessive deposits, wear, corrosion, et cetera, which are attributed to the inhibitor.

3.11 Specification requirements. A blend of the inhibitor at its maximum allowable concentration in a representative fuel shall meet all of the requirements of each applicable specification when tested in accordance with 4.6.8. For example, to be qualified for use in a motor gasoline, a gasoline conforming to MiL-G-3056 shall continue to meet all applicable requirements of MiL-G-3056 after the maximum allowable concentration of the inhibitor is added.

3.12 Storage stability. After storage for 12 months in accordance with 4.6.9, the inhibitor shall show no precipitation, layering, or other evidence of gross separation or degradation. Inhibitor representing the top half of the stored sample shall meet all requirements of this specification except 3.10.

3.13 Induction system deposit. For use in motor gasolines conforming to V-G-1690 and MIL-G-3506, the inhibitor shall pass the induction system deposit test performed in accordance with 4.6.10.

3.14 Emulsification tendency. To obtain approval for use in motor gasolines conforming to VV-G-1690 and MIL-G-3056 and diesel fuel conforming to VV-F-800, the inhibitor shall pass the emulsification tendency test performed in accordance with 4.6.11.

3.15 Accelerated stability. To obtain approval for use in diesel fuels conforming to VV-F-800 the inhibitor shall pass the accelerated stability test performed in accordance with 4.6.12.

3.16 Identification qualification data. The following properties of the finished inhibitor shall be determined but not limited during qualification: density 15°C, viscosity at 37.8°C, flash point, neutralization number, pH, and type of metallic constituent, if present (see 4.6.13). The permissible production variation of individual properties will be established at the time of qualification by mutual agreement between the manufacturer and the qualifying activity. Individual batches of inhibitor subsequently subjected to qualification conformance inspections shall conform to the established range of properties. The ranges shall not adversely affect any of the inhibitor performance characteristics such as relative effective concentration and Water Separometer Index Modified.

3.17 Workmanship. The finished product in bulk or container shall be uniform in appearance and visually free from grit, undissolved water, insoluble components, or other adulteration. The material shall have no adverse effect on the health of personnel when used for its intended purpose. Evidence to this effect shall be subject to review by departmental medical authority (see 6.3).

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Classification of inspections. The inspection and testing of the fuel soluble inhibitor shall be classified as follows:

- a. Qualification inspection (see 4.3)
- b. Quality conformance inspection (see 4.4).

4.3 Qualification inspection. Qualification inspection and testing shall consist of tests specified under 4.6.

4.3.1 Test report. A certified test report shall be forwarded to the activity responsible for qualification before the qualification sample is supplied. The test report shall contain laboratory data showing the results required by 3.2, 3.4, 3.5, 3.7, 3.8, 3.9, 3.11, 3.12, and 3.16. The test report shall also contain laboratory data on any of the special tests conducted to qualify the inhibitor for use in motor gasoline and diesel fuel (e.g., 3.13, 3.14, and 3.15). In addition, complete formulation data shall be supplied to the qualifying activity. This data shall include chemical composition (I.U.P.A.C. nomenclature and structural diagrams of each ingredient), the percentages of each ingredient, the manufacturer and trade names of each ingredient, and where available, the purity of each ingredient. The contractor shall furnish toxicological data and formulations required to evaluate the safety of the material for the proposed use.

4.3.1.1 Qualification sampling. Unless otherwise specified by the activity responsible for qualification, an initial 1-liter sample of finished inhibitor shall be submitted for evaluation by all of the tests with the exception of the storage stability and aircraft turbine engine tests. If the product passes these tests, an additional sample of finished inhibitor will be requested for the storage stability and aircraft turbine engine tests. Samples shall be identified as required and forwarded to the laboratory responsible for testing as designated in the letter of authorization from the activity responsible for qualification (see 6.5).

4.3.1.2 Requalification. Requalification will be required in the event any change in composition or formulation, source of the inhibitor or its ingredients, or manufacturing sites is made.

4.3.1.3 Retention of qualification. The retention of qualification of products approved for listing on the qualified products list (QPL) shall be accomplished by a periodic verification to determine continued compliance of a supplier's product with the requirements of this specification. The verification intervals shall not exceed two years. Unless otherwise specified by the activity responsible for the qualified products list, verification of qualification may be made by certification.

4.4 Quality conformance inspection. Quality conformance inspection of a bulk lot of inhibitor shall consist of tests for conformance to requirements for solubility (3.3), ash (3.8), pour point (3.9), and property limits shown on the Qualified Products List. In addition, the product shall be required to pass a rusting test when blended in depolarized iso-octane at the relative effective concentration and tested in accordance with 4.6.3.1, and shall also be required to give a Water Separometer Index Modified of 70 or higher (average of three tests) when tested at the maximum allowable concentration in accordance with 4.6.4.

4.4.1 Inspection lot

4.4.1.1 Bulk lot. A bulk lot is defined as an indefinite quantity of a homogeneous mixture of material offered for acceptance in a single isolated container, manufactured as a single isolated batch, or manufactured by a single plant run (not exceeding 24 hours) through the same processing equipment with no change in ingredient material.

4.4.1.2 Packaged lot. A packaged lot is defined as an indefinite number of 55-gallon drums or smaller unit packages of identical size and type, offered for acceptance, and filled with a homogeneous mixture of material from a bulk lot.

4.4.2 Sampling. Each bulk or package lot of material shall be sampled for verification of product quality and compliance in accordance with ASTM D 270.

4.5 Inspection. Inspection shall be in accordance with Method 9601 of FED-STD-791.

4.6 Test methods

4.6.1 Other Inhibitors. The maximum allowable concentration of inhibitor shall be mixed with each of the following fuels. The fuel shall contain no other inhibitors. Immediately after mixing and at the end of 24 hours, the samples shall be visually inspected for precipitation, cloudiness, or other evidence of insolubility.

- a. JP-4 fuel conforming to MIL-T-5624 containing the maximum allowable concentration of inhibitor conforming to MIL-I-27686
- b. A motor gasoline conforming to MIL-G-3056, MIL-G-46015, or VV-G-1690
- c. A diesel fuel conforming to VV-F-800 or MIL-F-46162.

4.6.2 Compatibility

4.6.2.1 Inhibitors. Grade JP-4 fuel (MIL-T-5624) containing the maximum allowable concentration of the inhibitor under test and no other inhibitors shall be mixed in equal proportions with samples of MIL-T-5624, grade JP-4 fuel containing the maximum allowable amount of each inhibitor previously qualified under this specification. The MIL-T-5624, grade JP-4 fuel used shall contain the maximum allowable amount of inhibitor conforming to MIL-I-27686. At the end of a 24-hour period, the samples shall be visually inspected for precipitation, cloudiness or other evidence of noncompatibility.

4.6.2.2 Static dissipator additive. Grade JP-4 fuel (MIL-T-5624), filtered through clay as described in appendix A.4 of ASTM D2550, shall be blended with each static dissipator additive approved in MIL-T-5624 to provide test fuels having a conductivity of 400 picosiemens per meter (pS/m) \pm 100 pS/m. After a 24-hour period, to insure that equilibrium fuel conductivity has been established the inhibitor under test shall be added and mixed. At the end of another 24-hour period, no more than \pm 40 percent change in the electrical conductivity of the fuel shall have occurred as a result of the test inhibitor. The fuel electrical conductivity shall be measured using either ASTM D 2624 or ASTM D 3114 test methods. The post-test visual inspection of the sample shall reveal no precipitation, cloudiness or other evidence of noncompatibility. (NOTE: Some loss in fuel conductivity with time may occur when bare glass bottles or bare metal cans are used with fuels containing static dissipator additives. The use of an epoxy-coated container is suggested. Also, fuel conductivity is temperature sensitive; no significant change in temperature should be allowed during the test.)

4.6.3 Relative effective concentration. The relative effective concentration of the inhibitor shall be determined by testing the inhibitor at various concentrations in depolarized iso-octane in accordance with 4.6.3.1. The inhibitor shall be tested at concentrations divisible by 3 (e.g., 6, 9, 12, 15, ... 33, and 36 grams inhibitor per cubic meter of fuel). No intermediate concentrations shall be tested. The relative effective concentration shall be defined as the lowest concentration giving a passing result in accordance with 4.6.3.1.6.

4.6.3.1 Rusting test method

4.6.3.1.1 Test apparatus. The test apparatus shall conform to the following requirements:

- a. Oil bath, conforming to ASTM D 665, with the additional requirement that it must be capable of maintaining the test sample at a temperature of $38^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$
- b. Beaker, beaker cover, stirrer, stirring apparatus, and chuck and motors for holding and rotating specimens while polishing shall conform to ASTM D 665
- c. Infrared heat lamp, 250 watts
- d. Hypodermic syringe, glass, 30-ml, with 6-inch stainless steel needles
- e. Disposable microliter pipets, consisting of calibrated capillary tubes containing 1, 2, 3, 4, 5, 10, 15, and 20 microliters
- f. Column, chromatographic, glass, 40 mm ID \times 600 mm with poly (tetrafluoroethylene) stopcock. A separatory funnel, Squibb, 1-liter, with poly tetrafluoroethylene) stopcock may be substituted for the chromatographic column
- g. Specimen holder, poly (tetrafluoroethylene), dimensions as specified in ASTM D 665 for type 2 holder
- h. Specimen, dimensions as specified in ASTM D 665, made of grade 1018 steel in accordance with ASTM A 108. The specimen shall be fabricated from 0.625 inch diameter round stock by machining or grinding to the final diameter of 0.50 inch. The specimen may be reused from test to test but shall be discarded when the diameter is reduced to 0.375 inch.

4.6.3.1.2 Test materials. Test materials shall conform to the following requirements:

- a. Silica gel, 28-200 mesh, heated to 225°F for 2 hours and cooled in a desiccator before use.
- b. Test solvent, iso-octane conforming to TT-S-735, which has been freshly depolarized as follows: A glass chromatographic column or 1 liter separatory (Squibb) funnel is filled with silica gel to a height 20 cm above the stopcock, retaining the silica gel by means of a glass wool plug. (NOTE: Do not use stopcock grease). One gallon of iso-octane is passed through the silica-gel bed by gravity, discarding the first 50 ml and collecting the remainder in a chemically clean glass container. The depolarized iso-octane should be used within 1 week after treatment.
- c. Test water, type B medium hard, prepared as follows: Make up three stock solutions using ACS reagent-grade chemicals in distilled water. Each solution shall contain one of these chemicals 16.4 g/liter NaHCO_3 , 13.2 g/liter $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, or 8.2 g/liter $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. Pipet 10 ml of the NaHCO_3 stock solution into 800 ml of distilled water in a 1-liter volumetric flask, and shake vigorously. While swirling the contents of the flask, pipet 10 ml of the CaCl_2 stock solution and then 10 ml of the MgSO_4 stock solution into the flask, add distilled water to bring the volume to 1 liter, and mix thoroughly. The final blend shall be clear and free of precipitation.

- d. Isopropanol, ACS reagent grade.
- e. Glassware cleaning solution.
- f. Lintless paper tissue (NOTE: Cel-Fibe 1710 Wipes, available from Cell-Fibe, Milltown, New Jersey, are satisfactory).
- g. Abrasive cloth, 150-, 240-, and 400-grit metal working aluminum oxide abrasive cloth, closed coat on jeans backing. The abrasive cloth is available in rolls of 1-inch tape, the most convenient form for use in this test.
- h. Disposable vinyl gloves.

4.6.3.1.3 Specimen preparation. The specimen, whether new or reused from a previous test, shall be cleaned by solvent rinsing or brushing as needed to remove oily residues, loose rust, or foreign material. After this preliminary cleaning, the specimen shall be handled only with vinyl gloves. (NOTE: It is essential to avoid contamination of the specimen, particularly by perspiration residues, and care should be taken to avoid transfer of such contaminants to the specimen via the abrasive cloth or the lintless paper tissues.) The specimen shall then be ground successively with 150-, 240-, and 400-grit abrasive cloth while mounted in the chuck of the grinding and polishing apparatus and turned at 1700 to 1800 rpm, in accordance with the following procedures:

- a. Grind with 150-grit cloth to remove all defects, irregularities, pits, and scratches as determined by visual inspection. Old 150-grit cloth may be used to remove rust or major irregularities, but the grinding shall be completed with new cloth. Stop the motor and scratch the static specimen longitudinally with one pass of new 150-grit cloth, using light pressure so that visible scratches appear.
- b. Grind with 240-grit cloth, removing all marks from the 150-grit cloth, and finishing with new 240-grit cloth. Stop motor and scratch the static specimen longitudinally with one pass of new 240-grit cloth, using light pressure, so that visible scratches appear.
- c. Polish with 400-grit cloth by wrapping a strip of cloth halfway around the specimen and applying a firm but gentle downward pull to the ends of the strip and moving the strip slowly along the specimen. Shift the position of the abrasive cloth frequently to expose fresh abrasive to the specimen. Continue this procedure, using new strips of abrasive cloth as required, until all marks from the previous 240-grit operation have been removed and the surface presents a uniform appearance, free of longitudinal or spiral scratches, with all polishing marks appearing to be circumferential. The final passes along the specimen shall be made with fresh abrasive cloth.
- d. Remove the specimen from the chuck, wipe the lintless tissue, and store in a beaker of depolarized iso-octane in a desiccator containing silica gel or other noncorrosive desiccant until ready for use. The storage period in the iso-octane shall not exceed 7 days.

4.6.3.1.4 Preparation of test blend. The test blend shall be prepared in the test beaker, not more than 2 hours before the immersion of the specimen in the test blend. The test blend shall be prepared in accordance with the following procedure:

- a. Clean the test beaker with a suitable cleaning solution (see note below). Clean the stainless steel stirrer and methyl methacrylate beaker cover by rinsing in any suitable aliphatic hydrocarbon solvent such as a light naphtha or iso-octane, washing thoroughly with hot distilled water, and oven drying (not over 65.6°C for cover). NOTE: If a glass stirrer or beaker cover is used, it should be cleaned in the same manner as the test beaker. Any suitable cleaning method that provides cleaning quality comparable to the use of chromic acid may be used. The use of a detergent cleaning solution is suggested. Use stainless steel forceps to handle the glassware. Wash with tap water and then with distilled water. Rinse with reagent grade isopropyl alcohol and dry in the air or oven. Detergent cleaning avoids the potential hazards and inconvenience associated with handling corrosive chromic acid solutions. The latter remains as the reference cleaning practice and as such may function as an alternative to the preferred use of detergent solutions.
- b. Prepare the blend of iso-octane and inhibitor in the test beaker with direct addition of the inhibitor. No intermediate blends, concentrates, or stock solutions are permitted. Prepare each test blend using between 300 and 400 ml of iso-octane in the test beaker. Use pipet or pipets to add integral numbers of microliters of the inhibitor to the beaker to increase measurement accuracy. Add the calculated volume of depolarized iso-octane to the test beaker. Fill the appropriate microliter pipet or pipets with inhibitor, wipe off excess, and force the inhibitor into the iso-octane. Allow the pipet to fill with iso-octane by capillary attraction and force this rinse into the test beaker. Repeat the rinse four times. Calculate the amount of iso-octane and inhibitor to be added to the test beaker using the instructions given in 4.6.3.1.c.
- c. Calculate the volume of iso-octane required for each concentration desired using the following equation, where density is in g/ml at 15°C:

$$\text{ml of iso-octane} = \frac{(\text{density of inhibitor}) (\text{microliters of inhibitors}) (1000)}{(\text{desired inhibitor concentration, grams/cubic meter})}$$

For example, assume the inhibitor has a density of 0.95 and the desired concentration is 6 grams/cubic meter of fuel. Calculate the volume of iso-octane required when using 2 microliters of inhibitor:

$$\text{ml iso-octane} = (0.95) (2) (1000)/(6) = 316.7$$

For inhibitor having a density less than 0.9 g/ml, the volume of iso-octane for many concentrations of interest will be less than 300 ml or more than 400 ml. Use the following procedure:

- (1) Calculate the volume of inhibitor required for 300 ml of iso-octane.

- (2) Increase the volume of inhibitor to the next integral microliter and add to 300 ml of iso-octane in the test beaker. Mix well.
- (3) Calculate the amount of inhibitor/iso-octane blend to be removed from the test beaker to leave the desired amount of inhibitor.
- (4) Remove the calculated amount of inhibitor/iso-octane blend and replace with an equivalent volume of depolarized iso-octane. Mix well. This approach is illustrated for the above example:
 - (a) Using this equation, calculate the desired volume of inhibitor for 300 ml of iso-octane for an inhibitor with a density of 0.85 and for a desired concentration of 6 g/m³.
$$300 \text{ ml iso-octane} = (0.85) (X \text{ microliters inhibitor}) (1000)/(6),$$

$$X = 2.12 \text{ microliters of inhibitor}$$

- (b) Add the next integral volume of inhibitor (i.e., 3 microliters) to 300 ml iso-octane and mix well. This gives an inhibitor concentration of 3 microliters inhibitor/300 ml iso-octane or 1 microliter/100 ml.
 - (c) The desired amount of inhibitor is 2.12 microliters. Thus, we need $(2.12)(100) = 212$ ml of inhibitor/iso-octane blend.
 - (d) Remove 88 ml of the inhibitor/iso-octane blend (i.e., $300 - 212 = 88$ ml). Replace with 88 ml of depolarized iso-octane. Mix well. This results in the correct volume of inhibitor (i.e., 2.12 microliters) in 300 ml of iso-octane.
- d. Place the beaker in the oil bath which has been regulated previously to maintain a sample temperature of $38^\circ\text{C} \pm 0.5^\circ\text{C}$. The beaker is inserted in a hole of the bath cover and suspended at a level such that the oil level in the bath is not below the sample level in the beaker. Cover the beaker with the beaker cover and the stirrer in position. Adjust the stirrer so that the shaft is 6 mm (0.24 inch) off-center in the beaker, and the blade is within 2 mm (0.08 inch) of the bottom of the beaker. Then suspend a thermometer through the hole in the cover intended for that purpose, so that it is

immersed to a depth of 57 mm (2.2 inches). Stir for at least 5 minutes. Turn off the stirrer. Using a clean pipet or syringe, withdraw enough test blend to leave exactly 300 ml in the beaker. Allow the test blend to come to $38^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$. Replace the thermometer with a cork or plastic plug.

4.6.3.1.5 Test procedure. After preparing a test specimen as described in 4.6.3.1.3 and a test blend as described in 4.6.3.1.4, the test shall be performed in accordance with the following procedure:

- a. Remove a test specimen from the iso-octane in the desiccator and wipe dry with a lintless paper tissue, handling with vinyl gloves throughout this step and the following operations. Repolish with 400-grit abrasive cloth by wrapping a strip of the cloth halfway around the specimen and applying a firm but gentle downward pull to the ends of the strip. Move the strip slowly along the specimen, twice in each direction, shifting the strip after the first back-and-forth pass so that fresh abrasive is exposed to the specimen. Inspect the specimen to insure that the surface presents a uniform appearance, free of longitudinal or spiral scratches, with all polishing marks appearing to be circumferential. Additional polishing is required if the specimen appearance is other than described. After polishing is completed, remove the specimen from the chuck, wipe lightly with lintless paper tissue, and screw the specimen into the specimen holder. Rinse the specimen with a stream of isopropanol from a wash bottle. Wipe dry immediately, wiping twice with fresh lintless paper tissues, using firm pressure and rotating the specimen while drawing through the tissue. NOTE: Under conditions of high ambient humidity, it is necessary to heat the specimen to prevent condensation of moisture and premature rusting. Under such conditions before the rinsing operation, place the specimen and holder 6 inches from a 250-watt infrared heat lamp and rotate for 1 minute. Keep the specimen under the lamp while proceeding with the rinsing and wiping operations.
- b. Immediately after rinsing and wiping, insert the specimen and holder through the specimen hole in the beaker cover and suspend the specimen so that its lower end is 13 to 15 mm (0.51 to 0.59 inch) from the bottom of the beaker. Leave the specimen in the test blend for a 10-minute static soak, then turn on the stirrer and soak dynamically for 20 minutes. NOTE: When multiple tests are run simultaneously, it is permissible to extend the static soak period to not more than 40 minutes in the case of the "first-in" specimen, giving the "last-in" specimen a 10-minute soak.
- c. Turn off the stirrer. Remove the cork or plug from the beaker cover, and add 30 ml of test water to the test beaker, adding it very carefully to the bottom of the beaker by means of a hypodermic syringe. Change to a clean needle for each test beaker. Replace the cork or plug in the beaker cover.
- d. Start the stirrer immediately and run for 5 hours, holding the bath temperature at the same setting so that the test samples will be maintained at $38^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$.

- e. At the end of 5 hours, stop the stirrer, remove the specimen and holder, rinse immediately with isopropanol, and allow to air-dry. Examine at once without magnification under normal indoor illumination, approximately 60-foot candles, scanning the surface very carefully, to detect any small pits. Record observations of visible rust, pits, stains, or deposits.

4.6.3.1.6 Interpretation of test results. A test shall be reported as failing if the center 48mm-(1.875-inch) section of the specimen shows six or more rust spots of any size, or if it shows any rust spot 1 mm in diameter or larger. (NOTE: The ends of the specimen, outside the center section, are ignored in rating the specimen.) Visible deposits or stains other than rust shall not constitute failure; deposits or stains may be examined microscopically to determine their classification. In order to assign a pass-fail rating to a given inhibitor at a given concentration, two tests shall be performed. The inhibitor shall be reported as passing at the given concentration if both tests give passing ratings, or failing at the given concentration if both tests give failure ratings. If the two tests give one passing rating and one failing rating, two additional tests shall be performed. If either or both of these additional tests give a failing rating, the inhibitor shall be reported as failing at the given concentration. If both of the additional tests give passing ratings, the inhibitor shall be reported as passing at the given concentration.

4.6.4 Water separometer Index modified. The inhibitor shall be blended into the reference fluid base, as described in ASTM D 2550, and tested in accordance with ASTM D 2550. For any given concentration of inhibitor, the average of three tests results shall be used to determine the conformance to the requirements of 2.7 or 4.4. In qualification testing to define the maximum allowable concentration, the inhibitor shall be tested at one or more concentrations selected from the following: 9, 13.5, 18, 22.5, 27, 31.5, 36, 40.5, 45, 49.5, or 54 grams inhibitor per cubic meter of fuel. No intermediate concentrations shall be tested. Only the ASTM CRC Water Separometer apparatus shall be used for qualification testing.

4.6.5 Ash content determination. The ash content of the inhibitor shall be determined in accordance with ASTM D 432, using a platinum crucible.

4.6.6 Pour point determination. Pour point shall be determined in accordance with ASTM D 97.

4.6.7 Aircraft engine test. The engine shall be operated for 100 hours in accordance with the engine operating requirements of MIL-L-7808. Grade JP-4 fuel, conforming to MIL-T-5624 shall contain twice the maximum allowable concentration of the inhibitor. Upon completion of the test, components of the engine exposed to the fuel such as a fuel controls, fuel nozzles, combustion section, turbine blades, exhaust section, elastomers, fuel/oil heat exchangers, and fuel pumps shall be examined for evidence of excess wear, deposits, corrosion or other deleterious effects. This test shall be performed by the activity responsible for qualification (see 6.5).

4.6.8 Specification tests. The inhibitor shall be added at its maximum concentration to a base fuel that contains no inhibitor but is otherwise representative of each grade of fuel for which the additive is to be qualified. The blend of fuel and inhibitor shall be subjected to all of the tests of each applicable specification.

4.6.9 Storage stability test. Two 1-quart amber glass bottles shall each be filled with 850 ml of the inhibitor and shall be tightly capped by means of a screw cap having a conical polyethylene liner. Each bottle shall be wrapped in a minimum amount of opaque packing material sufficient for protection against mechanical damage, but minimal in thermal insulation qualities. The

wrapped bottles shall be enclosed in a tight wooden or metal box for further protection against breakage and sunlight. The crated samples shall be stored at ambient, outdoor conditions in a temperate climate. The box shall be kept off the ground and protected from direct sunlight and precipitation under a canopy, open shed roof, or similar ventilated shelter. The crated samples shall be stored undisturbed in an upright position for the specified period. One of the samples shall be stored for exactly 12 months and then removed for examination and testing; the other samples shall be stored for 12 months or less and may be removed for examination and testing at any time at the option of the qualifying activity. Whenever a sample is removed for examination and testing, it shall be uncrated with minimum disturbance; the bottle shall not be shaken, inverted, or otherwise agitated. The contents of the bottle shall be inspected visually for precipitation, separation into layers, or other evidence of gross separation. The presence or absence and the nature of such separation shall be recorded. The top half of the liquid sample shall be carefully removed by suction or siphoning into another bottle, without disturbing the bottom half of the original sample. The top-half sample, after transfer to the second bottle, shall be shaken thoroughly and then used in laboratory testing, performed in accordance with 3.12. The bottom-half sample, in the original storage bottle, shall be retained for examination and possible additional testing to detect changes caused by storage.

4.6.10 Induction systems deposit test. The inhibitor, at its maximum concentration, shall be blended into a MIL-G-3055 motor gasoline. The test fuel shall then be tested for the formation of induction system deposits in accordance with Method 500 of FED-STD-791. The naphtha-washed deposits shall not exceed 2 mg/100 ml of fuel. The MIL-G-3056 gasoline without the inhibitor shall also be tested in accordance with Method 500 of FED-STD-791 concurrently to define the level of deposition occurring as a result of the inhibitor.

4.6.11 Emulsification tendency test. The inhibitor, at maximum allowable concentration, shall be blended into a MIL-G-3056 motor gasoline and a MIL-F-46162 diesel fuel. Each test fuel shall then be examined for emulsification tendencies in accordance with Method 550 of FED-STD-791. Interface ratings in excess of three are evidence of unsatisfactory emulsification tendencies and shall not be allowed. The MIL-G-3056 motor gasoline and the MIL-F-46162 diesel fuel shall also be tested in accordance with Method 550 of FED-STD-791 to identify the quality of the fuels before the addition of the inhibitor.

4.6.12 Accelerated stability test. The test inhibitor, at its maximum allowable concentration, shall be blended into a diesel fuel (VV-F-800) that contains no additives. Each test fuel shall be tested for the formation of total insolubles in accordance with ASTM D 2274. The total insolubles shall not exceed 1.5 mg/100 ml. The diesel fuel without the test inhibitor shall also be tested in accordance with ASTM D 2274 concurrently to define the level of insolubles occurring without the presence of the inhibitor. (NOTE: A suitable reference diesel fuel for this evaluation is described in Method 341.4 of FED-STD-791).

4.6.13 Identification tests. Identification tests shall be conducted in accordance with the following methods:

Density at 15°C	ASTM D 1298 or pyrometer
Viscosity at 37.8°C	ASTM D 445
Flash Point	ASTM D 56
Neutralization number	ASTM D 604, total acid number

pH	On 0.10 - 0.11 g sample in 125 ml of ASTM D 664 titration solvent. Read the constant pH as defined in note 9 of ASTM D 664.
Metallic constituent	Emission spectrograph not applicable for materials with ash contents of 0.05 percent or lower.

5. PACKAGING

5.1 Packaging, packing, and marking. The packaging, packing, and marking shall be in accordance with MIL-STD-290. In addition, package units shall be labeled to the extent applicable in accordance with Manual L-1.

6. NOTES

6.1 Intended use. The inhibitors covered by this specification are used, when specifically authorized, in jet engine fuels for the prevention of corrosion in fuel handling, transportation, and storage equipment and to improve the lubricating qualities of jet fuels. Certain of the inhibitors are also used in automotive gasoline, diesel fuel, and related petroleum products.

6.2 Ordering data. Procurement documents should specify the following:

- a. Title, number, and date of this specification
- b. Quantity required
- c. Selection of applicable levels of packaging and packing (5.1).

6.2.1 Amount of use of inhibitor. When Government procurement documents specify the use of inhibitors in fuels and related petroleum products, the concentration of inhibitor shall be specified in grams of inhibitor per cubic meter of fuel and shall not be less than the minimum effective concentration nor more than the maximum allowable concentration as listed on the Qualified Products List. Since the inhibitor is intended for use under many different environments, it is not possible to establish a single optimum concentration for all uses. Therefore, when a specific concentration is not required by the Government, the quantity of inhibitor used may vary to meet specific conditions.

6.3 Toxicity. Questions pertinent to toxicity should be referred by the procuring activity to the appropriate departmental medical service who will act as an advisor to the procuring activity. In case of Army procurement, the Surgeon General will act as advisor to the procuring activity.

6.4 Inhibitor for addition to fuels. When a fuel contractor or the Government purchases the inhibitor for addition to fuels to be used by the Government, the manufacturer of the inhibitor must certify to the purchaser that the product is an inhibitor that has been qualified under this specification. In addition, a test report showing compliance of the product with the requirements of 4.4 must be supplied to the purchaser. Additional data may be required by the purchasing activity to establish compliance with this specification.

6.5 Qualification. With respect to products requiring qualification, awards will be made only for products which are at the time set for opening of bids, qualified for inclusion in the applicable Qualified Products List whether or not such products have actually been so listed by that date. The attention of the contractors is called to these requirements, and manufacturers are urged to arrange to have the products that they propose to offer the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or orders for the products covered by this specification. The activity responsible for the Qualified Products List is the Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories (POSF), Wright-Patterson AFB, OH 45433, and information pertaining to qualification of products may be obtained from that activity.

6.6 Changes from previous issue. Asterisks are not used in the revision to identify changes with respect to the previous issue, due to the extensiveness of the changes.

Custodians:

Army — ME
Navy — AS
Air Force — 11

Preparing activity:

Air Force — 11

Project 6850-0597

Review activities:

Army — MD, AV
Navy — SH
Air Force — 68

User activity:

Army — AT

APPENDIX F
RPHPLC CHROMATOGRAMS OF CORROSION INHIBITOR IN CT JP-4

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a. DCI-4A
39.1 ppm

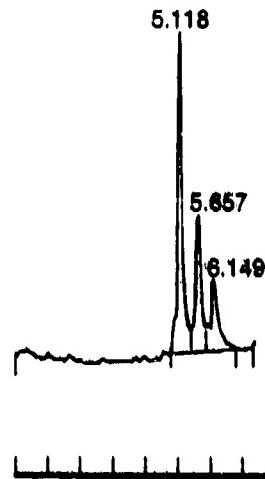


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. DCI-4A
26.3 ppm

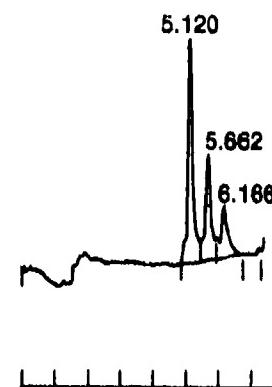


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. DCI-4A
13.3 ppm

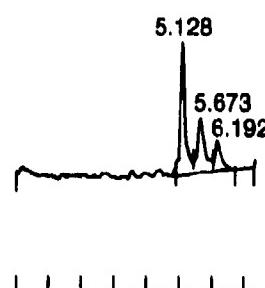


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. DCI-4A
6.6 ppm

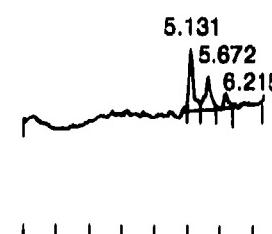


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346168

Figure F-1. — DCI-4A

a. DCI-6A
48.3 ppm

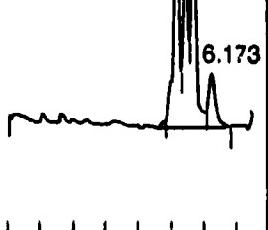


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. DCI-6A
24.6 ppm

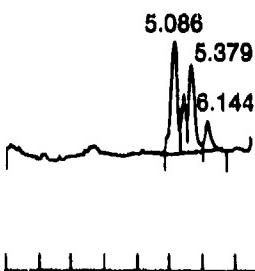


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Atten: 32 Zero: 10% 1 min/tick

c. DCI-6A
12.4 ppm

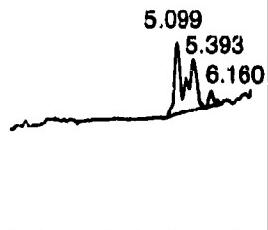


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. DCI-6A
6.3 ppm

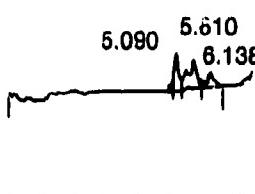


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346169

Figure F-2. -- DCI-6A

a. HITEC E-580
46.5 ppm

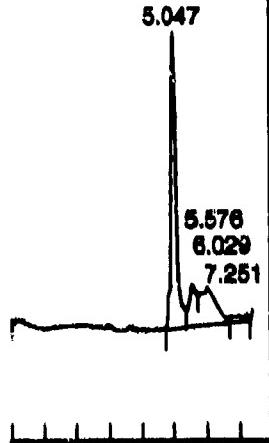


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. HITEC E-580
23.7 ppm

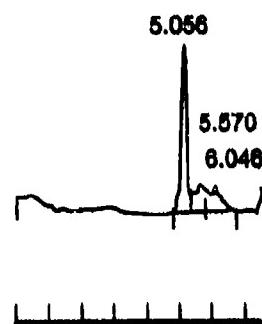


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. HITEC E-580
12.0 ppm

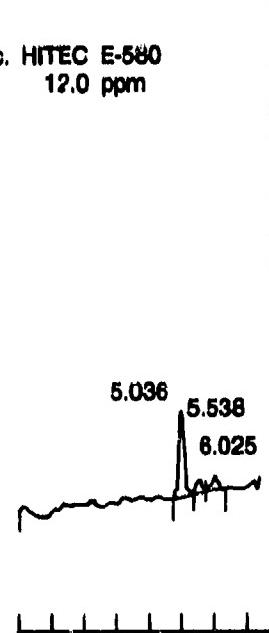


Chart Speed 0.9 cm/min
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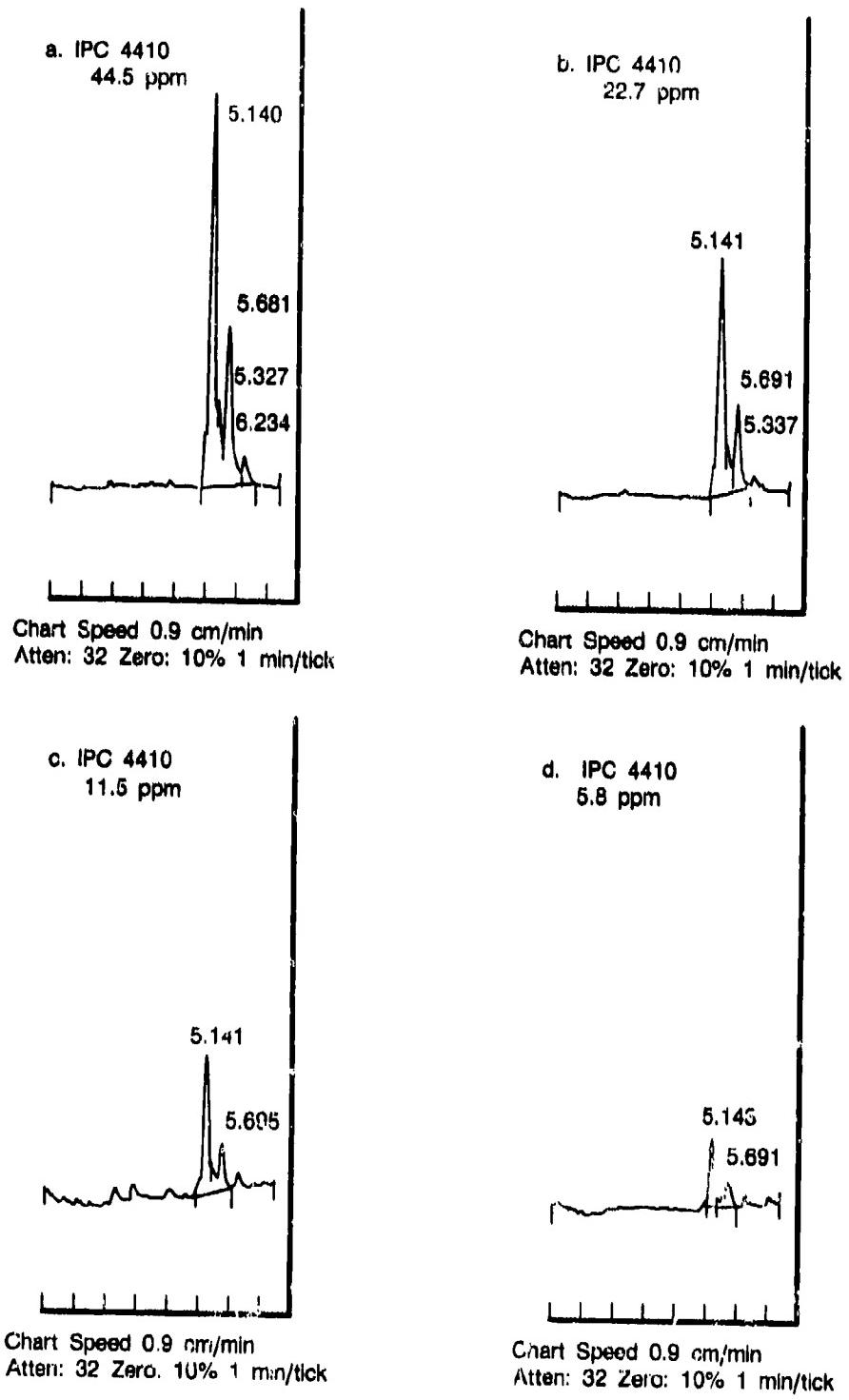
d. HITEC E-580
6.0 ppm



Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346170

Figure F-3. HITEC E-580



FDA 348171

Figure F-4. — IPC 4410

a. IPC 4445
41.8 ppm

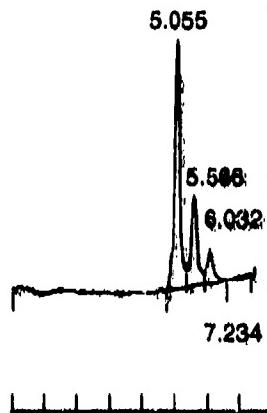


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. IPC 4445
21.3 ppm

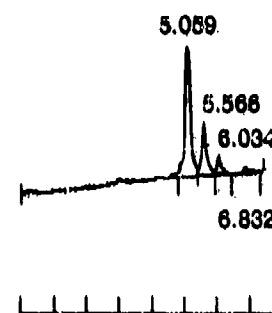


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. IPC 4445
10.8 ppm

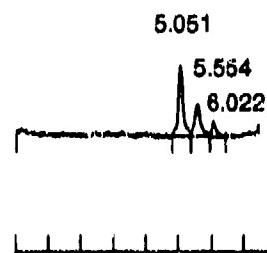


Chart Speed 0.9 cm/min
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d. IPC 4445
5.4 ppm

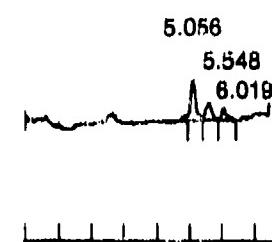


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346172

Figure F-5. -- IPC 4445

a. NALCO 5403
40.8 ppm

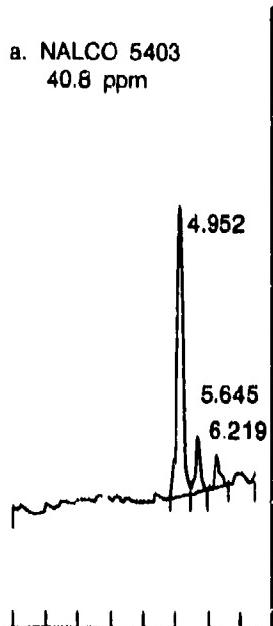


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. NALCO 5403
20.8 ppm

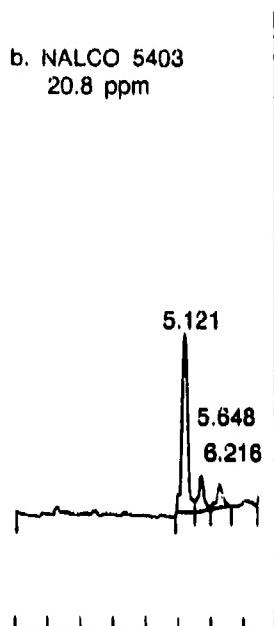


Chart Speed 0.9 cm/min
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c. NALCO 5403
10.5 ppm

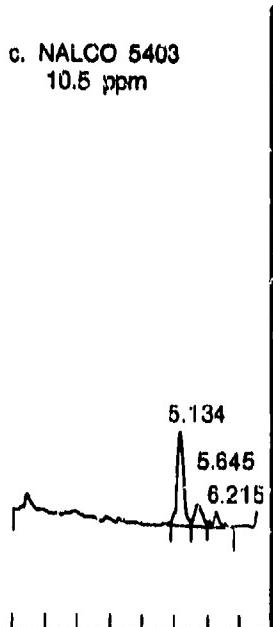


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. NALCO 5403
5.3 ppm

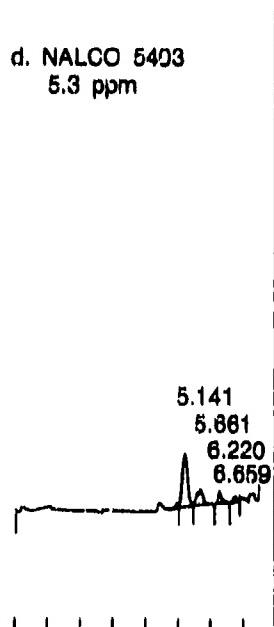
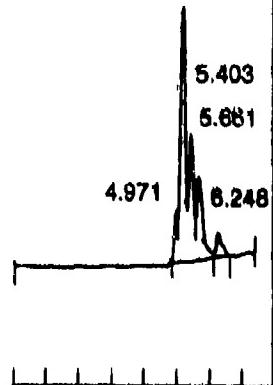


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346173

Figure F-6. — NALCO 5403

a. NALCO 5405
36.2 ppm



b. NALCO 5405
27.4 ppm

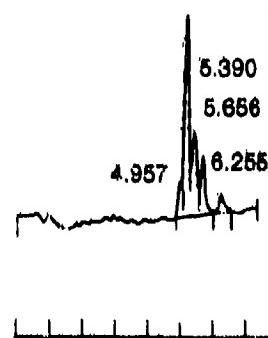
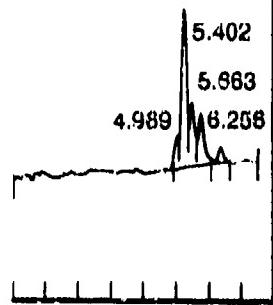


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. NALCO 5404
18.4 ppm



d. NALCO 5405
9.3 ppm

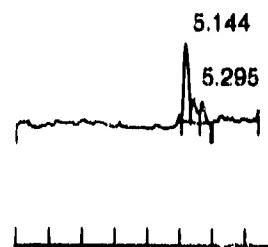


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 348174

Figure F-7. — NALCO 5405

a. NUCHEM PCI 105
42.1 ppm

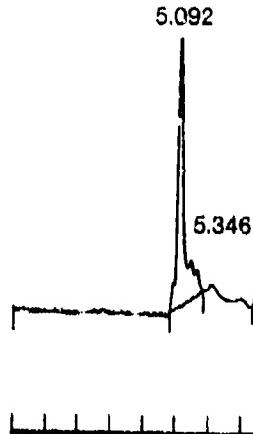


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. NUCHEM PCI 105
31.9 ppm

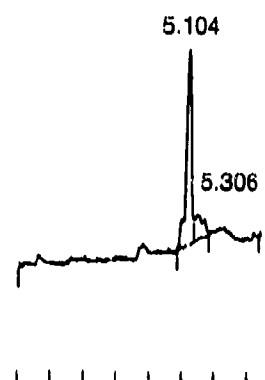


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. NUCHEM PCI 105
21.5 ppm



Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. NUCHEM PCI 105
10.8 ppm

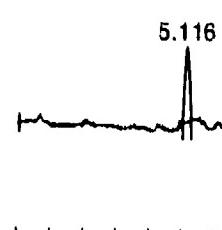


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346175

Figure F-8. — NUCHEM PCI 105

a. PRI-19
34.6 ppm

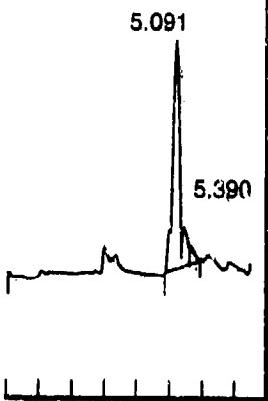


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. PRI-19
17.7 ppm

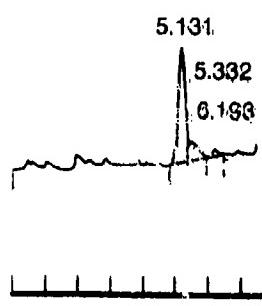


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. PRI-19
8.9 ppm

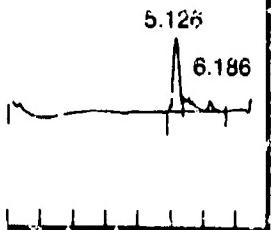


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. PRI-19
4.5 ppm

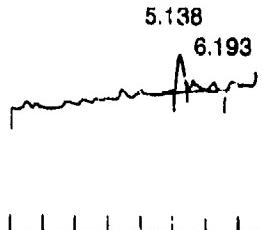


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346176

Figure F-9. — PRI-19

a. TOLAD 245
43.1 ppm

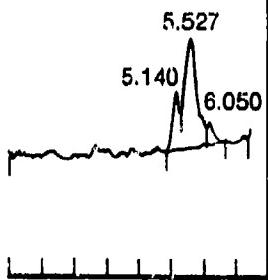


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. TOLAD 245
22.0 ppm

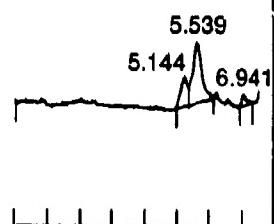


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. TOLAD 245
11.1 ppm

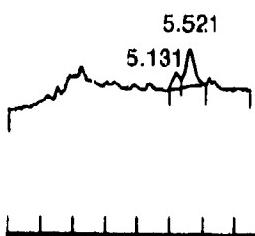


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. TOLAD 245
5.6 ppm

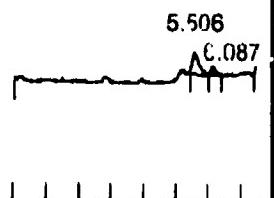


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346177

Figure F-10. — TOLAD 245

a. TOLAD 249
48.5 ppm

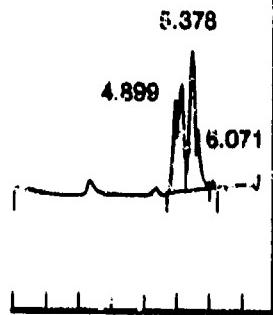


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. TOLAD 249
24.7 ppm

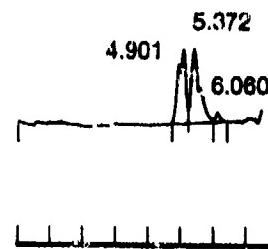


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. TOLAD 249
12.5 ppm

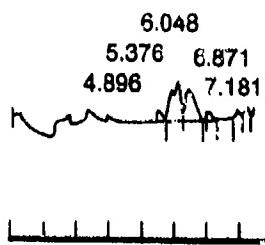


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. TOLAD 249
6.3 ppm

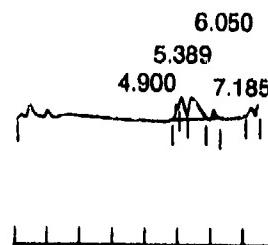


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346178

Figure F-11. — TOLAD 249

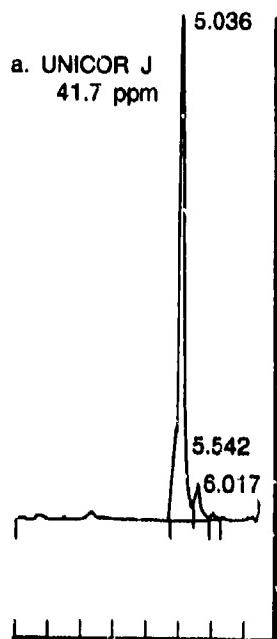


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

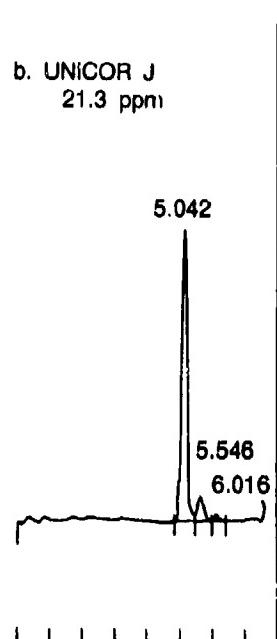


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

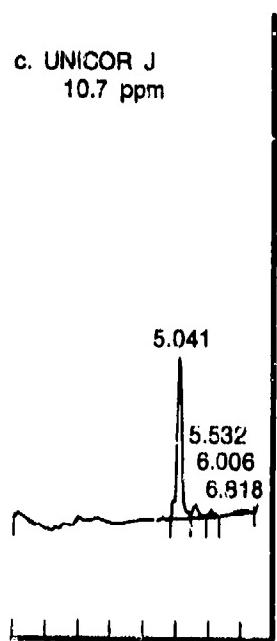


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

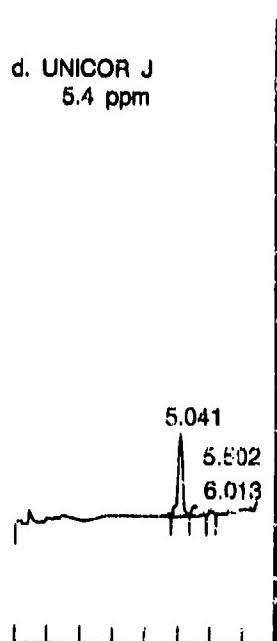
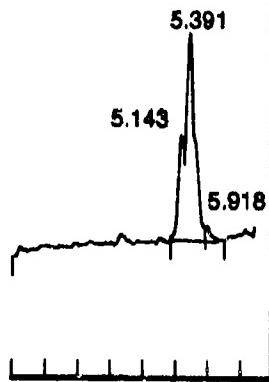


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 248183

Figure F-12. — UNICOR J

a. LUBRIZOL 541
40.6 ppm



b. LUBRIZOL 541
20.7 ppm

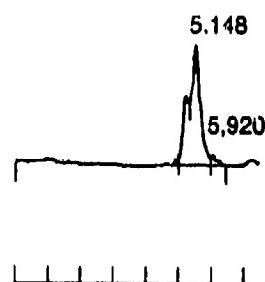


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. LUBRIZOL 541
10.5 ppm

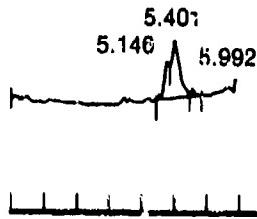


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. LUBRIZOL 541
5.2 ppm

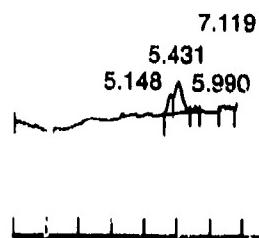


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346184

Figure F-13. — LUBRIZOL 541

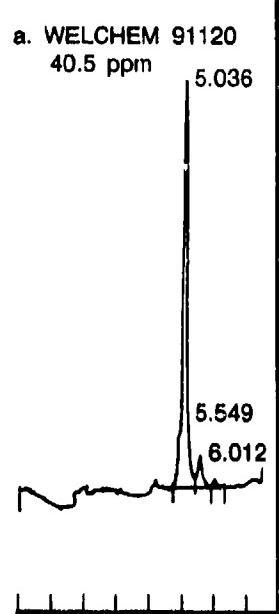


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

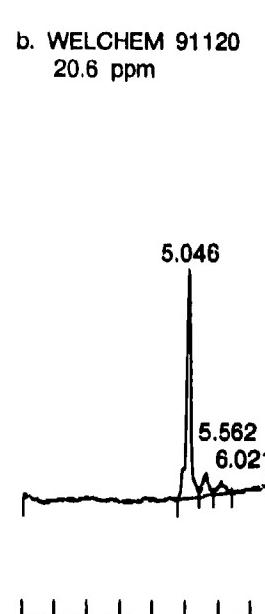


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

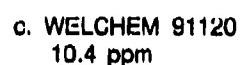


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick



Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346185

Figure F-14. — WELCHEM 91120

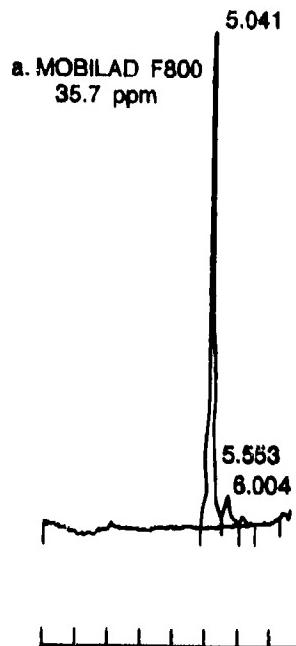


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. MOBILAD F800
24.0 ppm

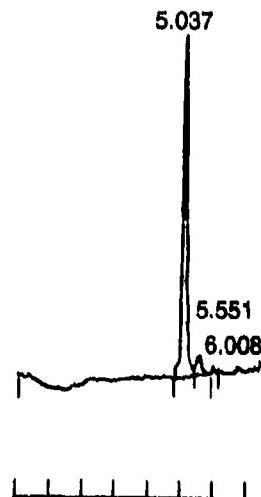


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. MOBILAD F800
12.2 ppm

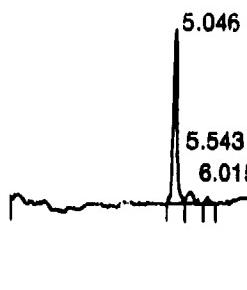


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

d. MOBILAD F800
6.1 ppm

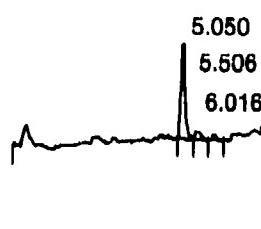


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 346180

Figure F-15. — MOBILAD F800

a. JP-4 (No CI)

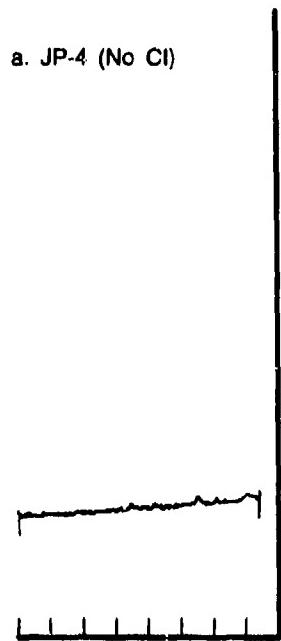


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. SUN A/F JP-4



Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

FDA 340829

Figure F-16. -- Clay Treated JP-4 and Additive Free JP-4

a. Trimer
33.8 ppm

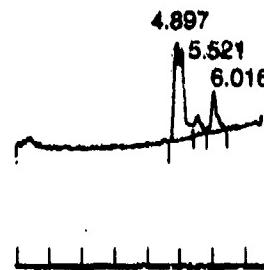


Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

b. Dimer
36.3 ppm

5.044
5.531
6.010

Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

c. Monomer
39.6 ppm

5.549

6.004
5.037

Chart Speed 0.9 cm/min
Atten: 32 Zero: 10% 1 min/tick

DA 340830

Figure F-17. — Trimer, Dimer, and Monomer Linoleic Acids